Analytical Abstracts

A monthly publication dealing with all branches of analytical chemistry: issued by the Society for Analytical Chemistry

Volume 7
No. 5, Abstracts 1637–2030

1960

Published for the Society by
W. HEFFER & SONS LTD., CAMBRIDGE, ENGLAND

Binding . . .

W. Heffer & Sons Ltd. announce that they are now able to BIND copies of ANALYTICAL ABSTRACTS, in an approved binding case, at a cost of 14/6 per volume. The 12 Parts for 1959 together with Index and remittance for 14/6 should be sent to

W. HEFFER & SONS, LIMITED
HILLS ROAD, CAMBRIDGE

IMPORTANT NOTICE TO SUBSCRIBERS

(Other than Members of the Society)

The Subscription to *Analytical Abstracts*, inclusive of Index, is £5 per annum; and for *Analytical Abstracts* together with *The Analyst* and Indexes is £8 8s. per annum.

The price of single copies of *Analytical Abstracts*, including back numbers, is 10s. 6d. post free.

Analytical Abstracts is available printed on one side of the paper only, suitable for incorporation into reference systems; the cost is £5 7s. 6d. per annum, exclusive of Index.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 14, Belgrave Square, London, S.W.1. Telephone: BELgravia 3258.

Research by Soviet EXPERTS . . . Translated by Western Scientists

INSTABILITY CONSTANTS OF COMPLEX COMPOUNDS

by K. B. Yatsimirskii and V. P. Vasil'ev

Instability constants quantitatively characterize the equilibrium in solutions of complex compounds and, in this connection, are very widely used by chemists in different fields (analytical chemistry, electrochemistry, technology of nonferrous and rare metals, etc., for appropriate calculations.

The instability constants of 1381 complex compounds are given. The authors have prefaced the summary of instability constants with an introductory section which examines methods of calculating instability constants from experimental data, the effect of external conditions (temperature and ionic strength) on the stability of complexes and the main factors determining the stability of complex compounds in aqueous solutions.

Cloth **200 pages** **$6.75**

SOVIET RESEARCH ON COMPLEXES AND CO-ORDINATION CHEMISTRY

This collection consists of 372 articles by Russian scientists in the general area of the chemistry of complexes. These reports have been complied from the Consultants Bureau translations of the following Soviet journals:

Journal of General Chemistry
Journal of Analytical Chemistry
Bulletin of the Academy of Sciences
Division of Chemical Sciences
Proceedings of the Academy of Sciences
Journal of Applied Chemistry
Soviet Journal of Atomic Energy

The articles have been grouped into five volumes, each of which may be purchased separately.

I. Inorganic Complexes—103 articles $65.00
II. Metal-Organic Complexes—86 articles $45.00
III. Thermodynamic and Kinetic Studies—57 articles .. $37.50
IV. Complexes in Analytical Chemistry—57 articles .. $37.50
V. Condensed Inorganic Systems; Miscellaneous —38 articles .. —31 articles .. $45.00

Complete collection .. $200.000

Tables of contents on request

Payment in sterling may be made to Barclays Bank in London, England

CONSULTANTS BUREAU ENTERPRISES, INC.
227 West 17th Street · New York 11, N.Y., U.S.A.

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1637. **Analysis for industry [redox titrimetry].** A. M. G. Macdonald. *Ind. Chem.*, 1959, **35**, 293–296; 505–507.—Reviews of recent advances are presented. (106 references.) S.C.I. ABSTR.

1638. **Qualitative micro-analysis and chemical microscopy.** H. Waldmann (Chem. Res. Div., F. Hoffmann-La Roche & Co. A.-G., Basel). *Chimia*, 1959, **13** (7), 224–230.—A brief description is given of the principles and techniques of qual. analysis on the micro scale. Typical spot reagents for metals and for organic functional groups are discussed, and an outline of the possibilities of chemical microscopy for qual. analysis is given. H. M.

1639. **Quantitative trace analysis and chromatography.** H. Gubser (Anal. Dept., Ciba A.-G., Basel, Switzerland). *Chimia*, 1959, **13** (8), 245–248.—The importance in trace analysis of establishing the specificity and limit of sensitivity of the reaction in use and the limiting dilution for its utility is stressed. The use of oxidation, by O in an ignition tube or by $Mg(NO_3)_2$, is advocated to concentrate or solubilise trace elements. Blank determinations and direct comparisons with standards correct automatically for errors often sustained when a calibration curve is used. The methods for the trace analysis of Se (20 to 1000 p.p.m. in 50 mg with diphenylhydrazine), of P [4 to 400 p.p.m. in 50 mg with $(NH_4)_6Mo_7O_{24}$] and of Fe^{2+} (2 to 100 p.p.m. in 1 g with 1:10-phenanthroline) are briefly indicated. The use of paper chromatography in the determination of impurities in dyestuff intermediates is also discussed, particularly of isomeric naphthylaminesulphonic acids, 2-hydroxy- and 2:6-dihydroxyanthraquinone, and salts of R and G acids. J. P. STERN

1640. **Micro-analysis and applied psychology.** H. Gysel (Anal. Dept., Ciba A.-G., Basel, Switzerland). *Chimia*, 1959, **13** (8), 253–257.—Unsuspected individual errors, particularly in the estimation of the last place of decimals in micro-weighing, are observed. Errors are greatly reduced by a new technique for estimating this place, involving a comparison of the distances to the nearest division on both sides of the pointer. J. P. STERN

1641. **Systematic scheme of qualitative analysis for anions. IV.** I. K. Taimni and Manohar Lal (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1959, **21** (2), 105–109.—The previously described procedure for the qual. analysis of anions (*cf. Anal. Abstr.*, 1958, **5**, 2077) relies, in part, on digestion of the sample with Na_2CO_3 soln. and analysis of the clear liquid obtained on filtration. It is shown that a number of cations pass into this filtrate, some completely and some partially. The scheme is expanded to permit the detection of these. T. R. ANDREW

1642. **Systematic study of insoluble substances. IV. A simplified scheme for the detection of all types of insoluble substances.** I. K. Taimni and I. S. Ahuja (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1959, **21** (2), 141–146.—After the preliminary digestion of the sample with Na_2CO_3 soln. and filtration, the ignited residue is fused with a mixture of KOH, K_2CO_3 and KNO_3 (composition not given), cooled, extracted with *N* K_2CO_3 and filtered. The behaviour of a number of compounds under this treatment is listed, and a scheme of qualitative analysis is presented. T. R. ANDREW

1643. **Analysis by phosphor poisoning.** A. Bradley and N. V. Sutton (Associated Nucleonics, Inc., Garden City, N.Y.). *Anal. Chem.*, 1959, **31** (9), 1554–1558.—The ions Cu^{2+}, Ag^{+}, Hg^{2+} and Pt^{4+} reduce the luminescence efficiency of a cadmium sulphide phosphor, probably by substitution of surface Cd^{2+} to give opaque surface layers. This effect may be applied to the analysis of ions in soln., and is independent of the vol. of the soln. The determination of Cu in the range 5 to 20 μg can be made with an accuracy of $\pm 10\%$. As the effects produced by the poisoning are additive, a separation of the metals being determined is not possible, but the method can be used for the quant. scavenging of these ions from soln. J. H. WATON

1644. **Use of organic reagents in inorganic analysis. IX. Analytical reactions of maleanilic acids.** Sachindra Kumar Datta (Victoria College, Coochbehar, India). *Z. anal. Chem.*, 1959, **168** (6), 418–424 (in English).—The analytical behaviour of maleanilic acids prepared by treating maleic anhydride with aniline (**I**), *o*-chloro- (**II**), *p*-chloro- (**III**), 2:5-dichloro-, *m*-nitro- (**IV**) or *p*-nitro-aniline (**V**), *o*- or *p*-toluidine, *o*- or *p*-anisidine, *p*-phenetidine, *p*-aminoacetanilide (**VI**), 1- or 2-naphthylamine, *o*-(**VII**) or *m*-phenylenediamine, benzidine (**VIII**) or tolidine has been studied. The physical properties of the compounds and their reactions with various metals are tabulated. Most of the compounds except those from **I** to **VIII** gave quant. ppt. with Th and Zr. Incomplete pptn. were given with Hg^{+}, Pb and Ce^{4+}; Fe^{3+} gave a grey turbidity, but Fe^{3+} gave colorations from brown to violet which were unstable on standing or heating; Au and Pt were reduced. The anilic acid from **VIII** was insoluble in all common solvents and could not be studied. S. M. MARSH

1645. **A study of the stability of dithizone and silver dithizonate in carbon tetrachloride by the use of radioactive silver-110.** Nobuo Suzuki (Chem. Dept., Fac. of Engng, Tohoku Univ., Katabira-cho, Sendai). *Japan Analyst*, 1959, **8** (6), 349–353.—

From investigations on the extraction of labelled silver soln. it was found that dithizone in CCl_4 (10^{-5} to 10^{-6} *M*) is completely decomposed within one month. Irradiation with light increases the rate of decomposition. Silver dithizonate is stable in concn. $>10^{-5}$ *M* in CCl_4 in the presence of excess of dithizone, but in a concn. $<10^{-6}$ *M* it decomposes almost completely within one day. The purification of CCl_4 on a laboratory scale has almost no effect on the stability. K. SAITO

1646. **Origin and development of the use of acid - base indicators.** F. Szabadváry (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (3), 253–259 (in German).—A review is presented of the development of the application of natural acid - base indicators in qualitative and quantitative analysis, the introduction of synthetic indicators, the measurement of pH by colorimetric methods and various theories of indicator mechanism. (31 references.) S. M. MARSH

1647. **Nitron hydrochloride as an extraction indicator in the titration of acids in coloured solutions.** A. S. Babenko (Chernovits State Univ.). *Zavod. Lab.*, 1959, **25** (6), 653–655.—Nitron hydrochloride free from excess of acid is prepared as follows. A soln. of nitron (2 to 3 g) in a small excess of 3% acetic acid soln. is diluted to between 100 and 150 ml and shaken with an excess of 20% KI soln. The ppt. is collected and washed with cold water (0° to 2°) and then stirred with an excess of a suspension of AgCl. After 10 to 15 min. on a boiling-water bath, the hot mixture is filtered and the crystals of nitron hydrochloride that separate from the cooled filtrate are collected. The soln. in water shows a change from colourless to yellow between pH 6·59 and 8·12. Extraction titrations for acid are carried out by adding three to five drops of the saturated aq. soln. of the indicator, together with 3 ml of $CHCl_3$, to 10 ml of the acid soln. and titrating with alkali in the usual way until the end-point is approached, when vigorous shaking is used. G. S. SMITH

1648. **Erio green, a new adsorption indicator in argentimetry.** F. Ícha and M. Valenta (School of Agric., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (7), 2424–2425.—A 0·5% soln. of Erio green B (C.I. Acid Green 16) (**I**) was used for the argentimetric determination of I^-, Br^- and SCN^- and for the titration of Ag^+ with I^-. The addition of 3 to 5 drops of **I** soln. is recommended for a 10-ml vol. of the sample. The sharpness of the end-point can be increased by the use of a mixed indicator (0·5 g of **I** and 0·05 g of methyl orange in 100 ml of H_2O). The presence of Ca, Ba, Mg, Zn, Al and Cr^{III} salts causes no interference. J. ZÝKA

1649. **Investigation of some Variamine blue derivatives applicable as redox indicators.** L. Erdey, É. Bányai and É. B.-Gere (Inst. Gen. Chem., Tech. Univ., Budapest, Hungary). *Talanta*, 1959, **3** (1), 54–63 (in German).—A detailed electrochemical investigation of substituted derivatives of Variamine blue (C.I. Azoic Diazo Component 35) (**I**) for use as indicators with different electrophilic and nucleophilic groups is described. The reversible redox process of these compounds corresponds to a two-electron reaction and their oxidation mechanism to that of **I**. By means of the equations given, the redox potentials at desired pH values can be calculated and the points of inflection of the E_0' - pH curves give the acid - base dissociation constants of the oxidised and reduced forms. Nucleophilic substituents raise, and electrophilic substituents lower, the basicity of the **I** molecules. G. P. COOK

1650. **Thermometric titrations of acids in the presence of hydrolysable cations.** F. J. Miller and P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1498–1500.—Milli-equiv. amounts of free acid are determined in the presence of hydrolysable cations thermometrically. The temp., as measured by a thermistor, is plotted mechanically against the vol. of base added, the end-point being the intersection obtained by extrapolating the two straight-line portions of the curve. Methods are given for the determination of free acid in HF soln. of Zr, H_2SO_4 soln. of U^{VI} and Cu, and HNO_3 soln. of Th and U^{VI}. J. H. WATON

1651. **Visual titrations in non-aqueous solvents with benzanthrone as internal indicator.** Ram Chand Paul, Jaswant Singh and Sarjit Singh Sandhu (Dept. of Chem., Punjab Univ., Hoshiarpur, India). *Anal. Chem.*, 1959, **31** (9), 1495–1498.—Benzanthrone is studied as an indicator in the neutralisation of certain Lewis acids and bases in non-aq. solvents. The indicator shows reversible behaviour in the solvents $POCl_3$, $SOCl_2$ and SO_2Cl_2, but can only be used quantitatively in $AsCl_3$ when the base is used as titrant. No reversibility occurs in the non-polar solvents CCl_4 and chlorobenzene, and no quantitative use is possible. This behaviour of benzanthrone serves to underline the fundamental difference between polar and non-polar solvents. J. H. WATON

1652. **New method for the standardisation of hypobromite.** G. S. Deshmukh and S. V. Tatwawadi (Hindu Univ., Benares). *Z. anal. Chem.*, 1959, **168** (6), 411–414 (in English).—A colorimetric method for the standardisation of hypobromite, with $K_4Fe(CN)_6$ as primary standard, has been developed. The method is based on the oxidation of ferrocyanide by hypobromite in the pH range 8 to 8·5 (bicarbonate buffer) and measurement of the extinction of the yellow ferricyanide soln. obtained at 420 to 450 mμ, which is then compared with that of standard soln. Only dil. soln. of ferrocyanide and hypobromite that are almost colourless can be titrated. Results compare favourably with those obtained by amperometric titration, with sodium arsenate as standard. S. M. MARSH

1653. **Preparation of standard solutions of manganese(III) by the reduction of permanganate with hydrogen peroxide.** Isamu Tsubaki (Gov. Ind. Res. Inst., Saida-cho, Ikeda, Osaka). *Japan Analyst*, 1959, **8** (5), 318–319.—By the use of the stoicheiometric amount of H_2O_2 for the reduction—

$2MnO_4^- + 4H_2O_2 + 8H^+ = 2Mn^{3+} + 8H_2O + 4O_2$,

a standard soln. of Mn^{III} can be prepared in 1·5 *N* HCl, H_2SO_4 or H_3PO_4. The $KMnO_4$ soln. is titrated with a standard soln. of H_2O_2—

$2MnO_4^- + 5H_2O_2 + 6H^+ = 2Mn^{3+} + 8H_2O + 5O_2$,

and a four-fifths equivalent of the H_2O_2 is added to another portion of the $KMnO_4$ soln. to form the manganic salt. K. SAITO

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

1654. **The effect of dissolved oxygen on alternating current polarography of some metal ions.** Hideko Shirai (Inst. Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *Japan Analyst*, 1959, **8** (5), 311–314.—With a.c. polarography in KCl soln., the wave height of Cd decreases in the presence of dissolved O, to an extent that increases with increase in concn. of KCl, whilst that in an ammonium salt soln. does not. The extent of decrease in wave height in potassium salt soln. decreases in the sequence NO_3^-, Cl^-, Br^- and I^-. The effect of O is overcome by the use of an acid buffer such as K H phthalate, which increases the wave height of Zn and In. It would appear that the buffer action of NH_4^+ or phthalate prevents the increase in concn. of OH^- near the electrode.
K. Saito

1655. **Polarographic determination of micro amounts of copper, lead and zinc in the presence of iron.** A. A. Sakharov (All-Union Sci. Res. Inst. for Methods and Technique of Prospecting). *Zavod. Lab.*, 1959, **25** (7), 791–792.—For the determination of Cu and Pb in the presence of Fe, the soln. (1 to 1·5 ml) is treated with 150 mg of K Na tartrate and then with aq. NH_3 to give a clear soln. After the addition of 50 mg of Na_2SO_3 and 5 to 10 mg of metol, aq. NH_3 is added to give a pH of $\simeq$10 (thymol blue paper), the soln. is diluted to 2 ml and the polarogram for Cu and Pb over the range −0·2 to −0·8 V is recorded. In this basal soln. the $E_{\frac{1}{2}}$ *vs.* the N.C.E. are −0·45 and −0·6 V, respectively. For the determination of Pb and Zn, the soln. (2 ml, *N* in HCl or H_2SO_4, containing $\lt$0·05 mg of Fe per ml) is treated with 50 to 60 mg of $NaHCO_3$ added in small portions, then with 50 mg of ascorbic acid, and finally with $NaHCO_3$ to give a pH of between 5 and 6 (alizarin red S paper). The ascorbic acid reduces Fe^{III} and also dissolved O, for which reaction Fe is a catalyst. Polarograms between −0·2 and −0·6 V and between −0·8 and −1·3 V are recorded after 10 to 20 min. (the $E_{\frac{1}{2}}$ for Zn is −1·05 V).
G. S. Smith

1656. **Volumetric determination of copper, lead and cadmium in pyrites, marcasite and their ash by sodium diethyldithiocarbamate.** K. Lasiewicz and H. Zawadzka (Inst. of Sulphuric Acid and Phosphate Fertilisers, Luboń, Poland). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 1033–1039 (in Polish).—Sodium diethyldithiocarbamate (**I**) produces with Cu a brown ppt. which is soluble in $CHCl_3$; the brown soln. is titrated with $HgCl_2$ soln. until colourless. All the metals that react with **I** interfere and should be masked with EDTA (disodium salt). When working with pyrites and ash, Pb cannot be masked completely, but when determining 1 to 3 mg of Cu, up to $\simeq$6 mg of Pb can be tolerated, although it is better to separate Pb as $PbSO_4$. With Pb, **I** forms a white complex, which is extractable with $CHCl_3$. By adding a known amount of Cu, Pb is displaced, and the brown soln. is titrated with $HgCl_2$ soln., as described above. Both the complexes **I** - Pb and **I** - Cd are pptd. quantitatively from a soln. containing 0·25% of KCN, but only **I** - Pb is pptd. from 2% KCN soln.; Cd is obtained by difference.
W. B. Miaskowski

1657. **Analysis of zinc, lead and copper-base alloys.** P. T. Beale (Platt Metals, Ltd., Enfield, Mddx., England). *Metal Ind., Lond.*, 1959, **95** (7), 139–140.—The application of the Hilger direct-reading attachment is examined and discussed, and the most sensitive lines for Cd, Sb, As, Fe, Mg, Sn, Bi, Ni, Cu, Zn, Pb, Al and Hg, with a condensed-spark discharge, are given. Modification of the electrodes enables them to be precisely mounted in the horizontal spark stand. Procedures and limits of determination for various elements are given for lead and lead alloys, zinc and zinc alloys and copper alloys. It is suggested that by the employment of a more arc-like discharge, as little as 0·01% of Pb and Zn could be determined in tin bronzes and that by the use of solution methods alloys of high Pb content, *e.g.*, leaded gun-metals, could be analysed.
S. C. I. Abstr.

1658. **Electrolytic separation of indium, thallium, zinc and cadmium and their determination in a single sample.** M. G. Sayun and P. P. Tsȳb (All-Union Sci. Res. Mining Metallurgy Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (7), 793–795.—The removal of As, Sb, Sn, Se and Te from a sample soln. containing In and Tl and various other metals is carried out by evaporation with HCl in the presence of hydrazine and KBr. The soln. is then treated with 10 ml of dil. H_2SO_4 (1 : 1) and evaporated to fuming, and the residue is dissolved in 30 to 40 ml of water; $PbSO_4$ is filtered off and washed with dil. H_2SO_4, the filtrate is adjusted with NaOH soln. to a pH between 2 and 3, and electrolysis is carried out at 50° in the cell described previously (*cf.* Sayun *et al.*, *Anal. Abstr.*, 1959, **6**, 3899) at a c.d. of 120 to 150 mA per sq. cm until Na is deposited on the mercury cathode (change of potential to −1·50 V). The excess of H_2SO_4 that is formed during the electrolysis is periodically neutralised with Na_2CO_3. After 2·5 to 3 hr. the amalgam is removed and decomposed electrolytically in 40 to 50 ml of *N* H_2SO_4 at 50°, with an anode potential not more positive than −0·47 V. When this potential is attained, the electrolysis is stopped and the Zn in the soln. is determined by the ferrocyanide method. The amalgam (containing Cd, In and Tl) is decomposed electrolytically in 40 to 50 ml of *N* H_2SO_4 at 50°, the electrolysis being stopped when the anode potential attains + 0·1 V. The electrolyte is separated and treated with 2 ml of 1·5% $FeCl_3$ soln., aq. NH_3 is added to precipitate the hydroxides of In and Fe, the ppt. is re-dissolved, and In is determined polarographically. Cadmium and Tl are determined polarographically in the electrolyte after the removal of In and Fe.
G. S. Smith

1659. **Colorimetric determination of hydrogen dissolved in water.** R. H. Robinson and D. B. Conklin (Atomic Power Div., Westinghouse Electric Corp., Pittsburgh 30, Pa., U.S.A.) *Anal. Chem.*, 1959, **31** (9), 1598–1599.—The method described by Stahlberg and Isoniemi (*Suomen Kem., B*, 1954, **27**, 86) is extended to cover the concn. range 5 to 50 ml of H per kilogram of H_2O. A known excess of $KMnO_4$ soln., containing NaH_2PO_4 to stabilise the pH, is added to the sample, which is then heated for 25 min. at 75°. The extinction of the soln. is read at 350 mμ, and is compared with that for $KMnO_4$ alone. On replicate samples, the coeff. of variation is $\simeq \pm 5\%$.
J. H. Waton

1660. **Solid scintillation counting of hydrogen-3 and carbon-14 in paper chromatograms.** H. H. Seliger and B. W. Agranoff (Dept. of Health, Education and Welfare, Bethesda, Md., U.S.A.).

Anal. Chem., 1959, **31** (9), 1607–1608.—The chromatograms are sprayed with a saturated solution of scintillation-grade anthracene in benzene. In this way fine micro-crystals of anthracene are incorporated in the paper and give a large increase in counting efficiency of low-energy β-rays as compared with gas-flow counting. The sprayed strips are stable for weeks and can be counted with reproducible results. K. A. PROCTOR

1661. **Determination of the water content of aqueous mixtures.** S. R. M. Ellis, R. D. Garbett and H. N. Sadler (Chem. Engng Dept., Birmingham Univ., England). *Ind. Chem.*, 1959, **35**, 498–500.—The calcium carbide method of Klockmann (*ChemikerZtg*, 1952, **76**, 706) has been modified. The reaction bomb and shaker are described and the procedure is exemplified by the determination of H_2O in ethyl acetate (**I**) and chloral hydrate (**II**). When the pressure of acetylene becomes constant, the H_2O content is obtained from a graph of pressure *vs.* wt. of H_2O. For H_2O contents $<5\%$, the accuracy can be improved by taking larger samples. With **I** (10 g, containing 1 to 3% of H_2O), the sample is diluted with acetone, and the accuracy is $\pm 0{\cdot}005$ g. With **II** (10 g, containing 1 to 10% of H_2O), no solvent is used and the accuracy is $\pm 0{\cdot}004$ g. For 10 to 40% of H_2O, a 1-g sample is taken. With water contents $<1\%$, the accuracy decreases. S. C. I. ABSTR.

1662. **Reagents for the separation and determination of potassium, rubidium and caesium.** Tadashi Hara (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1959, **37** (2), 112–119 (in English).—A detailed study of the solubility data for difficultly soluble salts of these elements is described. The choice of reagent, based on a large solubility difference between two difficultly soluble salts, has led to the testing of reagents of high molecular weight that give ppt. containing a large number of alkali-metal atoms and with no water of crystallisation and that can be produced in non-aqueous media. As a result, specific reagents for separation and determination have been found. $Na_2AgBi(NO_2)_6$ for separation of Rb from K, and either $NaBiI_4$ or $KBiI_4$ in conc. acetic acid for the separation of Cs from Rb.
E. G. CUMMINS

1663. **Composition of the caesium salt formed by bismuth potassium iodide reagent.** Tadashi Hara (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1959, **37** (2), 120–125 (in English).—The determination of the three elements present, Cs by the hexanitrodiphenylamine method after the removal of Bi, Bi by iodimetry after the separation of the addition compound of 8-hydroxyquinoline and $HBiI_4$, and iodine by iodimetry, has shown the caesium salt to be $Cs_3Bi_2I_9$. E. G. CUMMINS

1664. **Determination of caesium with bismuth potassium iodide or bismuth sodium iodide reagent.** Tadashi Hara (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1959, **37** (2), 126–131 (in English).—Defects in the conventional $HBiI_4$ method for the determination of Cs may be easily avoided by substituting conc. acetic acid for water as solvent. Fundamental conditions for the precipitation are discussed and an indirect method for the volumetric determination of Cs is outlined.
E. G. CUMMINS

1665. **Determination of caesium in the presence of other alkali-metal elements.** Tadashi Hara (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1959, **37** (2), 132–138 (in English).—Mixtures of Cs with Rb (2 parts of Cs: 1 part of Rb), Li (1:3), K (1:10) and Na (1:10), can be determined with $NaBiI_4$ by the usual method. Larger quantities of alkali metals are removed before the final pptn. of Cs is carried out.
E. G. CUMMINS

1666. **A new colorimetric method for the determination of a small amount of caesium.** Tadashi Hara (Ishibashi Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res. Kyoto Univ.*, 1959, **37** (2), 139–144 (in English).—In this determination of microgram amounts of Cs in the presence of large amounts of Rb, the sample is treated with $HBiI_4$ to give a ppt. of $Cs_3Bi_2I_9$, which is dissolved in dil. HNO_3 and extracted with dithizone - $CHCl_3$ to give Bi dithizonate (absorption max. 490 to 505 mμ), the extinction of which is proportional to the concn. of Cs. Tabulated results show the influence of increasing amounts of Rb (Rb:Cs $>$ 200:1) on the apparent Cs content, and these can be used for the production of corrected calibration curves for the determination of Cs once the approx. proportion of Rb to Cs is known. By this method, 10 to 45 μg of Cs can be determined in the presence of <4 mg of Rb. E. G. CUMMINS

1667. **Analytical applications of chelating agents. XVII. Murexide as indicator in the volumetric macro- and micro-determination of copper with 1:2-diaminocyclohexane-NNN'N'-tetra-acetic acid.** F. Bermejo Martínez and R. Rey Mendoza (Lab. de Quím. Anal., Fac. de Cienc., Santiago de Compostela, Spain). *Inf. Quím. Anal.*, 1959, **13** (4), 91–93, 100.—This article has also been published, in German, in *Z. anal. Chem.*, 1959, **167**, 261 (*cf. Anal. Abstr.*, 1960, **7**, 373).

XVIII. Spectrophotometric and absorptiometric micro-determination of copper with AEGT [1:2-di-(2-aminoethoxy)ethane-NNN'N'-tetra-acetic acid]. F. Bermejo Martínez and M. Paz Castro. *Ibid.*, 1959, **13** (4), 94–100.—Copper is determined from the absorption in soln. of its complex with this reagent. The complex is formed in the molar ratio of reagent to Cu of 1:1; the colour is very stable and is measured in a Spekker absorptiometer with an Ilford red filter No. 608. Beer's law is obeyed within the range 20 to 500 μg of Cu per ml, and pH changes within the range 4·90 to 9·77 have little effect. Interfering cations include Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+}. L. A. O'NEILL

1668. **Rapid volumetric method for determining copper in a carbonatite ore.** F. v. S. Toerien (Rio Tinto Mineral Search of Africa (Pvt.) Ltd., P.O. Box 65, Phalaborwa, N.E. Transvaal). *Analyst*, 1959, **84**, 565–567.—The sample is digested with HNO_3, H_3PO_4 and $HClO_4$ until all S has been removed. The diluted digest is boiled to dissolve soluble salts, oxalic acid soln. is added in drops until the change of colour indicates complete reduction of $HMnO_4$, NaF is added to the hot liquid to precipitate Th, and then Na acetate soln. After thorough cooling, KI is added and the liberated iodine is titrated with standard $Na_2S_2O_3$ soln. The method is rapid and accurate and suitable for

routine analysis of samples containing 0·03 to 5% of Cu. Iron, up to 50%, does not interfere.
A. O. JONES

1669. **Complexometric determination of copper and manganese when present together.** I. M. Yurist and P. G. Shakhova. *Zavod. Lab.*, 1959, **25** (7), 799–801.—The sample (0·5 to 1 g) of mixed oxides or hydroxides of Mn and Cu, containing between 32·5 and 6·5% of Cu and 42·0 and 67·5% of Mn (obtained in the analysis of semi-conductors for temperature-sensitive resistances) is dissolved in 2 to 3 ml of dil. HCl (1:1) with the addition of 2 to 3 ml of dil. H_2SO_4 (1:1) and the soln., diluted with 10 to 15 ml of water, is evaporated to fuming. The residue is dissolved in water and diluted to 200 ml, and an aliquot (20 ml) is neutralised to methyl orange with 10% Na acetate soln., then treated with 50 ml of water, 10 ml of a buffer soln. (50 ml of *N* Na acetate and 35 ml of *N* HCl in 250 ml) and 0·4 g of murexide - NaCl (1:100), and titrated for Cu with 0·05 *N* EDTA (disodium salt). The titrated soln. is treated first with a mixture of 6 ml of 0·05 *M* $Na_2B_4O_7$ and 4 ml of 0·2 *M* H_3BO_3 and then with 10 ml of a soln. obtained by dissolving 20 g of NH_4Cl and 80 ml of aq.NH_3 in 1 litre of water. The orange soln. is titrated with 0·05 *N* EDTA to a permanent red end-point to give the content of Mn. Alternatively, Cu is determined by titration in the presence of the borate buffer soln., and Mn is determined in the titrated soln. after addition of the ammoniacal buffer soln.
G. S. SMITH

1670. **Rapid polarographic determination of low concentrations of silver in the presence of interfering elements.** Y. Israel and A. Vromen (Israel Mining Industries, Haifa, Israel). *Anal. Chem.*, 1959, **31** (9), 1470–1473.—The method described can be used to determine Ag rapidly and simply in the presence of Cu and other interfering elements. A supporting electrolyte of aq. NH_3, NH_4Cl and KCN is used with a simple manual polarograph. The galvanometer deflection is read at constant applied voltage at the limiting current plateau of the reduction wave. The results were more accurate than those obtained by conventional polarographic procedures.
K. A. PROCTOR

1671. **Volumetric determination of silver in cobalt - silver mixture.** R. L. Shrimal (Dept. of Chemistry, M. L. B. Coll., Gwalior, India). *Analyst*, 1959, **84**, 568–569.—The soln. of the mixture (as nitrates) is evaporated just to dryness to remove acid and the residue is dissolved in water containing 100-vol. H_2O_2. An excess of EDTA (disodium salt) soln. is added and the liquid is boiled and then cooled. It is then passed through a column of Amberlite IR-120(H) at the rate of 7 ml per min., and the column is thoroughly washed with water to remove EDTA and the cobalt complex. The silver is eluted from the column with hot 16% HNO_3 and determined by the Volhard method. The error is within ±0·5%, and Ag can be determined even when ≃3% is present in the mixture.
A. O. JONES

1672. **Solvent extraction by the use of radio-isotopes. I. Extraction of silver dithizonate with carbon tetrachloride and chloroform.** Nobuo Suzuki (Chem. Dept., Fac. Sci., Tohoku Univ., Katahira-cho, Sendai). *Japan Analyst*, 1959, **8** (5), 283–287.—The rate of extraction with CCl_4 increases with the use of an excess of reagent at a given concn. of H_2SO_4. With $CHCl_3$, the acid concn. that gives a complete extraction of Ag decreases with increase of the excess of reagent. The interference of Cl^- in H_2SO_4 is eliminated by an increase in acidity, whilst that of Br^- and I^- is not. The presence of HNO_3 and $HClO_4$ lowers the rate of extraction. Salts of weak acids, including borate, tartrate, citrate and acetate, do not vitiate the extraction provided that the acidity is adjusted. Copper can be masked by EDTA.
K. SAITO

1673. **Further developments in the determination of oxygen in beryllium by the micro vacuum fusion method.** E. Booth and A. Parker (U.K. Atomic Energy Authority, Res. Group, Woolwich Outstation, England). *Analyst*, 1959, **84**, 546–548.—No modification of the technique previously described (*Ibid.*, 1958, **83**, 241) is required for the analysis of beryllium containing >0·1% of O. If the sample wt. is increased to ≃50 mg and the wt. of platinum in the bath to 10 g, and if vacuum-melted Specpure tin is added to the crucible before its temp. reaches 600°, and again with the sample, the oxygen content of beryllium can be determined at the 0·01% level, the coeff. of variation then being ± 15 to 20%. The results agree satisfactorily with those obtained by a method depending on fast neutron activation. The smallest amount of O that can be detected with certainty is ≃20 p.p.m.
A. O. JONES

1674. **Direct routine determination of lime in magnesite and similar materials.** H. V. Thompson, A. Mayer, G. Padget, R. C. Chirnside and H. Bennett (Brit. Ceram. Res. Ass., Stoke-on-Trent, England). *Trans. Brit. Ceram. Soc.*, 1959, **58** (6), 353–386.—A collaborative study shows that the most rapid and reliable chemical method for the determination of 0·1 to 7% of CaO in magnesite and allied materials is a modification of the Flaschka and Jakobljevich procedure (*cf. Anal. Abstr.*, 1955, **2**, 2985). The sample is dissolved in conc. HCl, and aq. triethanolamine soln. (50%) is added to complex Fe, followed by an excess of 0·05 *M* EDTA (disodium salt). The soln. is then made strongly alkaline and the excess of EDTA (present as its Mg complex) is titrated with 0·05 *M* $CaCl_2$, with 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid (Patton and Reeder, *Anal. Abstr.*, 1956, **3**, 2982) screened with murexide as indicator. The reproducibility is ±0·03% for ≃3% of CaO in a 1-g sample. Three different flame-photometric procedures are fully described; a monochromator instrument ensures even greater accuracy than is attained by the chemical method. In the first procedure, interference by Al, Si, P and Zr is avoided completely by using an excess of Sr as internal standard; an acetylene flame is preferred to a propane one, and measurements are made at 555 mμ. The standard deviation is 0·05 for ≃3% of CaO (21 analyses). In the second method Li is used as internal standard (this also obviates interference by Fe and Mg) and 1 g of Al_2O_3 (as $AlCl_3$ soln.) to nullify the effect of Al on the intensity of the Ca line (6162 A). The line 5530 or 4226 A is selected for the third procedure; no internal standard is required because of the high stability of the flame-photometric system (glass-prism monochromator, photomultiplier and micro-ammeter) (*cf.* Brealey and Ross, *Analyst*, 1951, **76**, 334).
W. J. BAKER

1675. **Determination of small amounts of calcium and magnesium in metallic lead.** S. D. Gur'ev and N. F. Saraeva (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (7), 795–798.—The

sample (1 to 20 g) of lead containing <100 μg of Ca or <200 μg of Ca plus Mg is treated with mercury (2 g for each g of Pb) to give a homogeneous mass and then with 100 ml of boiling water. The soln. is covered and boiled for 30 min., 4 ml of dil. HCl (1:1) is added, the soln. is filtered after having being set aside for 10 min., and the amalgam is washed twice by decantation with hot water; it is then treated with 25 ml of hot 1% HCl, stirred for 10 min., washed again, and removed. The combined soln. and washings are evaporated to dryness, and the residue is dissolved in 15 ml of water. If the pH of the soln. after cooling is <3 the evaporation is repeated. The soln. is diluted to 20 ml and mixed with 3 ml of Na diethyldithiocarbamate soln. (2% for a 20-g sample, 1% for a 1 to 5-g sample) and traces of heavy metals are completely removed by repeated extractions with 10-ml amounts of $CHCl_3$ with decreasing amounts of carbamate soln. If the aq. soln. shows a cloudiness that does not disappear during the shaking with $CHCl_3$, one drop of conc. HCl is added. The aq. soln. is heated to remove the $CHCl_3$ and to decompose carbamate, and the cooled soln., which should be transparent, is used for the determination. *Procedure for Ca alone*—After the addition of 1 ml of *N* NaOH and 0·5 ml of murexide soln. (prepared as required by dissolving 14 g in 25 ml of water, filtering, and adding 58 ml of ethanol), titration is carried out with EDTA (disodium salt) soln. (0·001 *M* with amounts of Ca >200 μg, otherwise 0·0005 *M*). *Procedure for Ca plus Mg*—Ammoniacal buffer soln. (5 ml) and three drops of Eriochrome black T soln. are added, and the diluted soln. (50 ml) is titrated with 0·0005 or 0·001 *M* EDTA (disodium salt). *Colorimetric determination of Mg*—The sample (0·1 to 2 g) is dissolved in HNO_3, the soln. is evaporated with HCl, $PbCl_2$ is removed and, after extraction of heavy metals as diethyldithiocarbamates, Mg is determined by means of Titan yellow. G. S. SMITH

1676. **Simultaneous determination of calcium and magnesium by the EDTA titration method. I. Determination of calcium and magnesium in high-purity titanium slag.** Katsuhiko Naka and Hiromoto Iwamatsu (Osaka Titanium Co., Higashihama, Amagasaki, Hyogo-ken). *Japan Analyst*, 1959, **8** (4), 225–231.—The removal of Ti from an HCl soln. of titanium slag (containing 80 to 95% of Ti) is rapidly effected by the thiosulphate method (W. H. Thornton, "Titanium," 1927, p. 92), and the Ca and the sum of Ca and Mg ($>0·03$% of each) are titrated with EDTA, with 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid (**I**) (*cf.* Patton and Reeder, *Anal. Abstr.*, 1956, **3**, 2982) and Eriochrome black T (**II**) as indicators, respectively. Iron and Mn are removed with aq. Br soln. and the interference of Br is eliminated with $Na_2S_2O_3$. The time taken for the determination is $\simeq 4$ hr. *Procedure*—Fuse the sample (0·5 g) with NaOH (5 to 10 g), dissolve the melt in hot water (50 ml), neutralise with HCl and 20% NaOH soln. to methyl red. After the addition of HCl (1:3) (5 ml), dilute the product to 200 ml, boil with $Na_2S_2O_3$ soln. (20%) (5 ml) for 20 min. to precipitate the Ti, cool and dilute to 250 ml. Add HCl (1:3) (10 ml) to two 50-ml portions of the supernatant liquid, boil, make ammoniacal, add satd. aq. Br soln. (20 ml) and filter. Titrate the sum of Ca and Mg in one portion with 0·01 *M* EDTA in the presence of ammonium buffer (67·5 g of NH_4Cl and 570 ml of 28% aq. NH_3 soln. per litre) (10 ml), KCN soln. (5%) (5 ml) and **II** soln. (0·5 g of **II** and 4·5 g of hydroxyammonium chloride per 100 ml) (5 drops). For the determination of Ca, add NaOH soln. (20%) (15 ml) to the second 50-ml portion, decompose NH_4Cl by evaporating the soln. to 50 ml, neutralise with HCl (1:1) to methyl red, boil with HCl (1:1) (10 ml), neutralise with 20% NaOH soln., add 7 *N* NaOH (10 ml) and titrate with EDTA in the presence of **I** (0·2 g). K. SAITO

1677. **Determination of radioactive strontium.** A. S. Goldin, R. J. Velten and G. W. Frishkorn (Robert A. Taft Sanitary Engineering Center, Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1490–1492.—Low concn. of ^{89}Sr and ^{90}Sr can be determined in a wide variety of samples (water, soil and sludge, and biological material), and techniques for the removal of interfering substances such as Ca, Ba, Ra, polyvalent metals and phosphates are described. The ^{90}Sr is determined by β-counting the activity of ^{90}Y after its extraction into 2-thenoyltrifluoroacetone reagent, and the total activity is determined by β-counting a $SrCO_3$ ppt. K. A. PROCTOR

1678. **Back-titration of zinc with methyl red as adsorption indicator in the presence of ferrocyanide.** Takanosuke Nasu and Reiko Kumagai (Metal Engng Dept., Fac. of Engng, Tohoku Univ., Sakuraku-ji, Sendai). *Japan Analyst*, 1959, **8** (5), 306–311.—Tananaev's titration of zinc with $K_4Fe(CN)_6$ soln., with methyl red as adsorption indicator (*Z. anal. Chem.*, 1936, **107**, 92), is improved by a back-titration technique. When the titration is carried out at pH 1 with an excess of 0·1 *M* $K_4Fe(CN)_6$ at 60° to 70°, followed by back-titration with 0·15 *M* $ZnCl_2$ in the presence of 5 drops of ethanolic 0·1% methyl red soln., there is a sharp colour change from pale yellow to dark blue at the end-point. Magnesium and Sr interfere. K. SAITO

1679. **Separations involving sulphides. X. Separation of zinc from some elements that form thio salts.** I. K. Taimni and S. N. Tandon (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1959, **21** (2), 110–111.—By adding an excess of Na_2S soln., then acidifying with HCl, boiling and filtering off the pptd. sulphide, Zn, which remains in the filtrate, can be separated from binary mixtures with As, Sb, Te, Se, Mo, Hg, Au, Pt or Re. In the presence of As, Pt or Re, the final acidity before boiling should be 6 *N*, and *N* in the presence of the other elements. Notes on the separation are given. T. R. ANDREW

1680. **Determination of zinc in ores by the use of an anion-exchange resin.** Masanori Hisada and Kaichi Kashikawa (Akenobe Mine, Asako-gun, Hyogo-ken). *Japan Analyst*, 1959, **8** (4), 235–239.—Zinc and Cd are adsorbed on an anion-exchange resin from 0·12 *N* HCl containing 10% of NaCl, and are thus separated from Cu, Fe, Mn and alkaline-earth elements (Kallman *et al.*, *Anal. Abstr.*, 1956, **3**, 2013). Both Zn and Cd are eluted with ammoniacal NH_4Cl soln., the Cd is pptd. with Na diethyldithiocarbamate (**I**) and the Zn is titrated at 60° with EDTA (Eriochrome black T as indicator). Large amounts of Cu are masked with KCN and formaldehyde. The presence of $Fe(OH)_3$ in the eluate does not cause interference. *Procedure*—Dissolve the sample (0·5 to 1 g containing <50 mg of Cd) in HNO_3 (1:1) (20 ml), and heat to white fumes with H_2SO_4 (1:1) (10 ml) and conc. HBr (10 ml). Cool and dissolve the residue in water (50 ml), filter and evaporate to dryness. Dissolve

the residue in 0·12 *N* HCl containing 10% of NaCl (w/v) (50 ml), and pass the soln. through a column (diam. 2·5 cm) of Amberlite IRA-140 (40 to 60 mesh, 15 g, conditioned with the same soln.) (rate of flow, 5 ml per min.). Wash the column with the same HCl soln. and elute the Cd and Zn with *N* aq. NH_3 soln. containing 2% (w/v) of NH_4Cl. Add I (0·5 g), heat to 60°, add the indicator (4% in ethanol) (5 drops) and titrate with EDTA (1 ml ≡ 5 mg of Zn). K. SAITO

1681. **Determination of zinc in plant ash by oscillopolarography.** Katsumi Yamamoto (Chem. Dept., Fac. Sci., Tohoku Univ., Katahira-cho, Sendai). *Japan Analyst*, 1959, **8** (4), 245–248.—An oscillopolarograph was set up with a cathode ray tube oscilloscope, according to the Matheson and Nichol system (*Trans. Electrochem. Soc.*, 1938, **73**, 193), its sensitivity (2×10^{-4} *M* Zn) being superior to that of the Müller system (*Ind. Eng. Chem., Anal. Ed.*, 1938, **10**, 339). The ash (of *Prunus salicina* Lindl.) (1 g) is dissolved in HCl, the SiO_2 is removed with HF and HCl, and the Zn is extracted with dithizone in CCl_4. The Zn is back-extracted with 0·02 *N* HCl and diluted to 25 ml, and a 6-ml portion is evaporated to 1 ml, then mixed with NH_4Cl soln. (20%) (2 ml), aq. NH_3 soln. (6 *N*) (1·2 ml) and gelatin soln. (0·1%) (0·3 ml). The polarogram is recorded. K. SAITO

1682. **Complexometric determination of zinc and cadmium in [cadmium-based] solder.** E. Yu. Zel'tser. *Zavod. Lab.*, 1959, **25** (7), 798–799.—The sample (0·2 g) together with 0·1 g of tin is dissolved in 10 ml of dil. HNO_3 (1:1), the soln. is evaporated to 5 ml, 100 ml of hot water is added, and the soln. is filtered after 1 hr. The insol. matter is washed with 3% HNO_3, and the filtrate at 70° is treated dropwise with 2 *N* HCl until no more AgCl is pptd. The ppt. is filtered off, and the filtrate, after being made slightly acid to litmus paper, is treated with 10 ml of a buffer soln. (35 ml of aq. NH_3 and 5·4 g of NH_4Cl in 100 ml of water), then cooled, and titrated with 0·1 *M* EDTA (disodium salt) in the presence of Chromogene black ET-00 (C.I. Mordant Black 11) to the transition from violet to blue. This gives the content of Zn plus Cd. The soln. is then mixed with 50 ml of 5% Na diethyldithiocarbamate soln. to precipitate Cd, a further amount of the indicator is added, and the liberated EDTA is titrated in the presence of the ppt. with 0·1 *M* $MgCl_2$ to a violet end-point. The Zn is obtained by difference. G. S. SMITH

1683. **Analytical application of the hanging mercury drop electrode. Determination of impurities in high-purity zinc.** W. Kemula, Z. Kublik and S. Glodowski (Inst. of Phys. Chem., Polish Acad. of Sci., Warsaw Univ.). *J. Electroanal. Chem.*, 1959, **1** (1), 91–100.—Metallic impurities that form amalgams with mercury, and have reduction potentials more positive than that of Zn, can be determined. The simultaneous determination of six metals is described. The lowest concn. measured were—Pb, $6{\cdot}4 \times 10^{-5}$%; Cd, $8{\cdot}2 \times 10^{-6}$%; Sb, 2×10^{-5}%; and In, $6{\cdot}8 \times 10^{-4}$%. Details are given of the modification of the drop electrode described earlier (*cf.* Kemula and Kublik, *Anal. Chim. Acta*, 1958, **18**, 104). Separation procedures for metals with oxidation potentials close together are discussed. E. G. CUMMINS

1684. **Colorimetric determination of cadmium with Rhodamine B.** A. I. Lazarev and V. I. Lazareva (Kuibishev Industrial Inst.). *Zavod. Lab.*, 1959, **25** (7), 783–786.—In acid soln. (H_2SO_4), in the presence of KI, Cd gives a complex with Rhodamine B (C.I. Basic Violet 10) which shows a max. absorption at ≃550 mμ. The extinction varies linearly with concn. over the range 0 to 0·6 μg per ml. The molar extinction coeff. is $7{\cdot}4 \times 10^4$. To determine Cd in magnesium or aluminium, the sample (0·5 g) is dissolved in HCl with the subsequent addition of 1 ml of conc. HNO_3. The soln. is evaporated nearly to dryness, and the salts are dissolved in water (20 ml). The soln., in a separating-funnel, is mixed with 5 ml of 20% Na citrate soln. and 20 ml of water; a few drops of aq. NH_3 are added to give a neutral or alkaline reaction to Congo red paper, 5 ml of 0·5% Na diethyldithiocarbamate soln. is added to form complexes with Cd, Cu, Bi, Hg and Sb, and the complexes are extracted with 10 ml of CCl_4. The aq. soln. is treated with 2 ml of the carbamate soln. and again extracted with CCl_4 (5 ml); the extracts are washed with water, and the Cd is selectively re-extracted with 10-ml portions of *N* HCl. The combined aq. extracts containing the Cd are evaporated to dryness, the residue is dissolved in 2 ml of 3·6 *N* H_2SO_4, and the diluted soln. is treated with 2 ml of reagent soln. (obtained by dissolving 0·8 g of Rhodamine B in 40 ml of ethanol and diluting to one litre with water) and 2·5 ml of *N* KI, and diluted to 25 ml. In the presence of Fe^{III}, 1 ml of 2% ascorbic acid soln. is added after the Rhodamine B soln. The extinction is measured after 10 min. G. S. SMITH

1685. **Determination of cadmium in magnesium alloys by means of nickel diethylphosphorodithioate.** A. I. Busev and L. Ya. Polyak. *Zavod. Lab.*, 1959, **25** (6), 668–669.—The alloy (0·2 to 0·5 g) is dissolved in 15 ml of dil. H_2SO_4 (1:5) with the addition of a few drops of dil. HNO_3 or, if large amounts of Zr are present, 2 to 3 ml of HNO_3. The cooled soln. is treated with 15 ml of 0·05 *N* nickel diethylphosphorodithioate. The ppt. is collected after 1 hr., washed with a satd. aq. soln. of the cadmium salt, and dried to const. wt. Alternatively, the washed ppt. is dissolved in aq. NH_3, and the slightly acid soln. is titrated with 0·05 *N* iodine. G. S. SMITH

1686. **Rapid polarographic determination of low concentrations of mercuric ion.** Y. Israel (Israel Mining Industries, Haifa, Israel). *Anal. Chem.*, 1959, **31** (9), 1473–1475.—The method (*cf. Anal. Abstr.*, 1960, **7**, 1670) is rapid and accurate and is carried out with a manual polarograph. At a concn. of 1·0 m*M* Hg^{2+} the coeff. of variation (12 replicates) was ± 2·0% and at 0·01 m*M* it was ± 34% (12 replicates). The lowest concn. of Hg detectable was 5×10^{-3} m*M*. K. A. PROCTOR

1687. **Improved method for the determination of boron.** R. Wickbold and F. Nagel (Untersuchungslab. der Chem. Werke Hüls A.-G., Marl). *Angew. Chem.*, 1959, **71** (12), 405–406.—The "aspirating burner" of Wickbold (*Ibid.*, 1957, **69**, 530) has been applied to the determination of boron in boric acid and organic boron compounds. The sample is placed in a flask containing H_2SO_4 through which passes a rapid stream of N which leads into an oxy-hydrogen flame. When the sample is decomposed and any water present driven off, methanol is added and the flask is heated to 70° to 80°. On passage through the flame, methyl borate and other compounds are decomposed into B_2O_3, H_2O and

CO_2. The B_2O_3 is absorbed in water and determined volumetrically. After preliminary fusion of the sample with Na_2CO_3 the method can be applied to the determination of boron in glass. Recoveries of boron were within $\pm 0{\cdot}1$ mg of the theoretical for H_3BO_3, $Na_2B_4O_7$ and K and Na tetraphenylboron.
T. R. ANDREW

1688. **Determination of boric oxide in glass by pyrohydrolysis separation.** J. P. Williams, D. E. Campbell and T. S. Magliocca (Glass Res. and Develop. Div., Corning Glass Works, Corning, N.Y.). *Anal. Chem.*, 1959, **31** (9), 1560–1563.—The pyrohydrolytic determination of B_2O_3 is carried out in a platinum reactor tube at a temp. of 1350°. Uranium oxide (U_3O_8), mixed with a little $Na_2SiO_3 \cdot 9H_2O$, is used as accelerator. The distillate is titrated with 0·1 *N* NaOH in the presence of mannitol, as described by Webster (*Brit. Abstr. C*, 1952, 91). If F^- are present in the glass, 2 g of $CaCl_2 \cdot 2H_2O$ is added before the titration. Careful blank determinations are necessary. The results for B_2O_3 contents in the range 0·2 to 28% are $\simeq 0{\cdot}1\%$ (absolute) low compared with those obtained by conventional wet methods; more accurate results might be possible at a reactor-tube temp. of 1400°, or with different catalysts and operating conditions. In the presence of excessive amounts of Pb, Zn or P, inferior results are obtained. J. H. WATON

1689. **Analysis of sulphuric [aluminium] anodising solutions.** J. B. Mohler. *Metal Finish.*, 1959, **57** (8), 63–65.—It is shown that, with a mixture of methyl orange and phenolphthalein, free acid (H_2SO_4) is given by titrating to a faintly cloudy orange end-point, and total acid [H_2SO_4 plus $Al_2(SO_4)_3$ expressed as H_2SO_4] by titrating to a pink end-point. Using a pH meter with a glass electrode, the titration (*A*) to pH 3·6 gives the free acidity. Titration is continued to pH 8·9 (*B*), to give total acid. A further titration to pH 10·0 (*C*) corresponds to re-dissolution of $Al(OH)_3$. The ratio of $(C - A)$ to $(B - A)$ is 4:3, which provides a rapid check for errors. The two methods give comparable results. C. A. BLAU

1690. **Determination of oxygen and aluminium oxide in aluminium bronze.** R. Dufek and L. Kopa (Res. Inst. Metals, Panenské Břežany, Czechoslovakia). *Hutn. Listy*, 1959, **14** (7), 620–622.—The determination of oxygen and Al_2O_3, respectively, is based on the fact that Al dissolves in methanolic bromine soln. whilst Al_2O_3 remains undissolved and can be filtered off. The O is calculated from stoicheiometric relations. *Procedure*—To the sample (5 to 10 g) add methanol (200 ml) and bromine (10 ml) and dissolve carefully with gentle heating under reflux. To the yellow soln. add bromine (5 ml) and heat again. Add methanol (50 ml) and bromine (5 ml) and heat for 1 hr. Cool, filter twice through a filter-paper, wash the ppt. with methanol and several times with 0·1 *N* HCl. Ignite the filter in a platinum crucible at 1100°, fuse the residue with $KHSO_4$ (0·25 g), dissolve the melt in H_2O (50 ml) and transfer to a 100-ml flask; dilute to vol. with H_2O. To 10 ml add a 0·05% soln. of aluminon (15 ml), heat on a water bath for 5 min., cool, dilute to 100 ml and determine the Al content colorimetrically at 525 mμ. The results are in good agreement with those by the vacuum fusion method (*cf.* Sloman, *J. Inst. Metals*, 1945, **71**, 391) in a graphite crucible at 1650°. The aluminon soln. contains, in 1 litre, aluminon (0·5 g), Na acetate (25 g), glacial acetic acid (40 ml) and benzoic acid (1 g). J. ZÝKA

1691. **Complexometric titration of rare-earth elements. Dissolution of the rare-earth oxalate with EDTA and back-titration with magnesium sulphate.** Seizo Misumi and Tomitsugu Taketatsu (Chem. Dept., Fac. of Sci., Kyushu Univ., Hakozaki, Fukuoka). *Bull. Chem. Soc. Japan*, 1959, **32** (8), 873–876 (in English).—The conditions for this determination are studied. The rare-earth oxalate is pptd. with satd. oxalic acid soln., the crystalline ppt. is filtered off and dissolved in EDTA (disodium salt) soln. made alkaline with aq. NH_3, and the excess of EDTA is back-titrated with standard $MgSO_4$ soln. Rare-earth elements in concn. of 1 to 5 mg are determined rapidly with good accuracy. I. JONES

1692. **Spectrophotometric determination of lanthanum with neothorin [arsenazo].** Shozo Shibata, Fukuo Takeuchi and Teiichi Matsumae (Gov. Ind. Res. Inst., Nagoya, Japan). *Anal. Chim. Acta*, 1959, **21** (2), 177–181 (in English).—Lanthanum reacts with arsenazo (**I**) in basic soln. to give a red-violet water-soluble complex. The optimum wavelength for measurement is 570 mμ and the optimum pH is 9·0. Under these conditions with 2 ml of 0·1% aq. **I** soln. in 25 ml, 50 μg of La gives an extinction of 0·7 in a 1-cm cell. Citrate, tartrate, oxalate, phosphate and perchlorate must be absent. Up to 50 mg of acetate, sulphate, chloride or nitrate can be tolerated, and up to 500 μg of F^-. Many cations interfere. T. R. ANDREW

1693. **Preparation of carrier-free lanthanum-140 by electrolysis.** Hiroshi Hamaguchi, Nagao Ikeda and Toshi Kawashima (Fac. of Sci., Tokyo Univ. of Education, Koishikawa, Tokyo). *Japan Analyst*, 1959, **8** (6), 382–387.—When 0·1 *M* NH_4ClO_4 - 0·01 *M* $Ca(ClO_4)_2$ containing a ^{140}Ba - ^{140}La equilibrium mixture in perchlorate form is electrolysed at 6 V, the deposit of ^{140}La on a platinum cathode is almost radiochemically pure, irrespective of the duration of electrolysis and the pH (2·5 to 6·3). The radiochemical yield increases with time and reaches a max. (50%) after 30 min. A similar electrolysis is effected by the use of 0·25 *M* $(NH_4)_2SO_4$ of pH 3·5. The use of nitrate results in a change of pH during electrolysis and vitiates the radiochemical purity. This method can be satisfactorily applied to the separation of ^{90}Y from ^{90}Sr, the yield being $\simeq 80\%$ after electrolysis for 30 min. It is also applicable to the separation of ^{49}Sc from ^{49}Ca. K. SAITO

1694. **Neutron activation determination of impurities in silicon for semi-conductor material.** Toshio Nakai, Seishi Yajima, Isao Fujii and Minoru Okada (Japan Atomic Energy Res. Inst., Tokai, Ibaragi-ken). *Japan Analyst*, 1959, **8** (6), 367–372.—The neutron activation determination of various impurities in high-purity silicon was investigated with a boiling-water type of reactor (neutron flux 2×10^{11} per sq. cm per sec.), the elements being separated according to Kraus's method ["Int. Conf. on Peaceful Uses of Atomic Energy," Geneva, P/837 (1955)]. Because of insufficient density of the flux, only 0·03 to 0·5 p.p.m. of Cu and $\simeq 0{\cdot}01$ p.p.m. of Na could be determined; the lower limits of sensitivity for As, Sb, Cd, Zn, Ga and Fe were 4×10^{-3}, 1×10^{-3}, 2×10^{-2}, 1×10^{-1}, 3×10^{-2} p.p.m. and 4 p.p.m., respectively. K. SAITO

1695. **Spectrometric determination of silica in rocks, soil, plants and water.** M. Lheureux and J. Cornil (Lab. de Rech. Chim. du Min. de Congo Belge et du Ruanda-Urundi, Tervuren, Belgium). *Ind. Chim. Belge*, 1959, **24** (6), 634–640.—Alkaline orthosilicates prepared at a pH >10·9, when poured into an acid molybdate soln. at pH 1 having a ratio of H^+ to MoO_4^{2-} between 1·5 and 2 (expressed as equiv. per 100 ml), form a stable α-decamolybdosilicate. Molybdophosphate is extracted quant. with butanol - $CHCl_3$ (1:4) and the molybdosilicate is then extracted with butanol. *Procedure*—A pseudosoln. of the SiO_2 in HCl (0·1 to 2 mg in <25 ml) is neutralised with KOH and 0·5 ml of 10% KOH soln. is added. The soln. is added to a mixture of 10 ml of a 6% soln. of $Na_2MoO_4.2H_2O$, 10 ml of *N* HCl and 8 ml of butanol, and water is added to 100 ml. After shaking the soln. for 5 to 10 min., 5 ml of conc. HCl is added and the molybdophosphate is extracted with butanol - $CHCl_3$. The soln. is then re-extracted with butanol (2 × 10 to 15 ml), $(NH_4)_2S_2O_8$ (≃50 mg) is added to prevent reduction, and the butanol extract is diluted with 94% ethanol until it is clear and then made up to a known vol. with 30% ethanol. The extinction is measured at 400 mμ. The Beer - Lambert law is followed, and a calibration curve is prepared from pure quartz. This does not pass through the origin owing to traces of SiO_2 in the KOH. The method is used to determine the SiO_2 in the washings from a gravimetric determination after only one evaporation to dryness, and can be used directly for minerals low in SiO_2. In water, monosilicate can be determined separately by omitting the alkalisation stage, and the method is also applicable to vegetable ash. Interferences and errors are discussed. E. J. H. Birch

1696. **Analyses by the use of fluorotitanic acid and hydrogen peroxide. IV. Colorimetric determination of stannic tin.** Hisao Fukamauchi, Ryuko Enohara and Mitsuko Uehara (Tokyo College of Pharm., Ueno, Taito-ku, Tokyo). *Japan Analyst*, 1959, **8** (5), 315–317.—The extent to which the coloration of Ti with H_2O_2 is suppressed by F^- decreases with increase in concn. of Sn^{4+} and HCl. The working curve at 420 mμ is linear for <18 mg of Sn per 25 ml in 1 to 2 *N* HCl soln. The optimum molar ratio of the reagent soln. is Na_2TiF_6:NaF:H_2O_2 = 1:4:1 (2·08 g of Na_2TiF_6, 1·68 g of NaF and 1·1 ml of 30% H_2O_2 in 100 ml of *N* HCl). The reaction is complete almost instantaneously at room temp., and the colour remains unchanged for at least 1 hr. in a plastic apparatus; some kinds of plastic catalyse the decomposition of H_2O_2.
K. Saito

1697. **New gravimetric method for the determination of lead.** P. Ştefănescu and Y. Ştefănescu (Lab. Anal. Chim., Inst. "Petrochim," Ploieşti, Romania). *Stud. Cercet. Chim., Cluj*, 1959, **10** (1), 137–139.—Lead is pptd. by resorcinol. *Procedure*—The soln. of Pb^{2+} (20 to 30 ml) is treated dropwise in the cold with the reagent (a freshly prepared soln. of 10 g of resorcinol dissolved in 10 ml of 20% aq. NH_3 and diluted to 100 ml) until pptn. is complete, with an excess of 2 to 3 ml. When the supernatant blue liquid is clear, the ppt. is filtered off, with suction, in a G4 crucible, washed several times with H_2O, 5 or 6 times with ethanol, and 4 or 5 times with 2 to 3 ml of diethyl ether, then dried for 15 min. *in vacuo*. The conversion factor is 0·66032. Tin, Bi and Sb do not interfere. The determination occupies 30 to 45 min., and the errors are >0·3%. H. Sher

1698. **Micro-determination of lead and copper in solutions by a preliminary enrichment on an ion-exchange column.** V. B. Aleskovskiĭ, R. I. Libina and A. D. Miller. *Trudy Leningr. Tekhnol. Inst. Lensoveta*, 1958, (48), 5–11; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,207.—*Procedure*—Wash a column (20 mm in diam., 50 mm in height) filled with cation-exchange resin (1-mm mesh) with hot 15% HCl soln. until it is free from Pb and Cu. Pass the test liquid (*e.g.*, natural water) through at the rate of 0·6 to 1·0 litre per hr. Remove the adsorbed Pb and Cu from the resin by repeated treatment with hot 15% HCl for 5 min. After each acid treatment, decant the supernatant liquid on to a filter and wash the resin with 10-ml portions of hot H_2O. Evaporate the combined acid extracts and washings almost to dryness and determine Pb in the residue as the sulphide and Cu with dithizone. With 10 μg of Pb per litre the error is >20%. With 2 to 80 μg of Cu per litre the error is >25%. K. R. C.

1699. **Rapid polarographic analysis of lead and copper in commercial brass.** T. Capaccioli, W. Sbrolli and A. Vercellone (Soc. Bombrini Parodi-Delfino, Colleferro, Roma). *Ann. Chim., Roma*, 1959, **49** (7–8), 1125–1142.—To avoid the masking by Cu of the reduction step of small amounts of Pb, the Cu is pptd. as $Cu_2(SCN)_2$ with KSCN in the presence of hydroxyammonium chloride. The pptn. is carried out in the polarographic cell and the ppt. is not separated. The contents of Pb and Cu are derived from a comparison of the respective diffusion currents with that for the total metals present (in practice, Pb, Cu and Zn). The method is compared with a modification of that of Reynolds and Rogers (*cf. Brit. Abstr. C*, 1949, 194, 371).
L. A. O'Neill

1700. **Estimation of titanium by a.c. polarography.** J. R. Beevers and B. Breyer (Dept. of Agric., Univ. of Sydney, Australia). *J. Electroanal. Chem.*, 1959, **1** (1), 39–43.—A study of the behaviour of Ti^{IV} - Fe^{III} mixtures has led to the development of a method for the determination of Ti, which is satisfactory even in the presence of a large excess of Fe. Gelatin has been introduced to suppress the disturbing phenomena attributed to streaming of the electrolyte. The calibration curve shows that an Fe to Ti ratio of 75:1 can be tolerated in a supporting electrolyte of 0·2 *M* oxalic acid containing 5·0 × 10^{-3} % of gelatin. The titanium concn. range tested was 10^{-4} to 10^{-3} *M*.
E. G. Cummins

1701. **Determination of zirconium in rocks and quartz sands rich in iron.** M. Lucco Borlera (Turin Polytech., Italy). *Ric. Sci.*, 1959, **29** (7), 1465–1471.—The greater part of the Fe is eliminated by distillation in a stream of Cl in the presence of graphite at ≃350°. A second distillation at 650° permits the quant. separation of Zr as $ZrCl_4$. The Zr in the distillate is determined amperometrically by pptn. with cupferron, interference from residual Fe^{3+} being eliminated by reduction with ascorbic acid. The method is suitable for concn. of Zr of ≃0·1 to 1% and the error is <2% when the ratio of Fe to Zr is >25:1. Titanium interferes but Si does not, although it is preferable to concentrate the Zr, when present in small amounts, by removing the Si with HF. L. A. O'Neill

1702. **Determination of trace impurities in zirconium metal by a point-to-plane spectrochemical procedure.** R. F. Farrell, G. J. Harter and R. M.

Jacobs (Westinghouse Electric Corp., Pittsburgh, Pa.). *Anal. Chem.*, 1959, **31** (9), 1550–1554.—A rapid point-to-plane procedure is described for the determination of impurities in zirconium in the p.p.m. range; it may be extended to impurities and alloying elements in Zircaloy-2. Normal spectrographic procedures are used for recording and computing the results. The sample, after being ground to a 32-micro-inch surface, is subjected to a pre-burn of 10 sec., and then a total exposure of 1 min. An ignited a.c. arc (an a.c. arc combined with an a.c. spark) is used with 5 to 7 radio-frequency amp. The metals that may be determined are Al, B, Cd, Co, Cr, Cu, Fe, Hf, Mg, Mn, Mo, Ni, Pb, Si, Ti, V and W. Suitable solid standards are necessary. J. H. WATON

1703. **The separation of thorium(IV) and cerium(III) ions by using alginate as cation exchanger.** Takeo Takahashi and Shingo Miyake (Inst. of Ind. Sci., Univ. of Tokyo, Yayoi-cho, Chibo). *Bull. Chem. Soc. Japan*, 1959, **32** (8), 878–879 (in English).—The soln. containing Th^{4+} and Ce^{3+} (at pH 2 to 2·5) is added to the alginate (acid form) (prep. described), which is eluted first with 0·075 *N* HNO_3 and then with *N* HNO_3 to obtain Ce^{3+} and Th^{4+}, respectively. Colorimetric determinations are carried out on the eluates with H_2O_2 and arsenazo. I. JONES

1704. **X-ray fluorescence analysis of thorium oxide in monazite.** Tokunosuke Nakajima and Masao Takahashi (Japan Atomic Energy Res. Inst., Tokai, Ibaragi-ken). *Japan Analyst*, 1959, **8** (4), 249–253.—Carl's method (ASTM S. T. P. No. 157, 1953, 63) was examined with reference to the effect of grain size (optimum 250 mesh) and of U (tolerance limit, 2%) by the use of an X-ray Industrial Quantometer. For 0·75 to 10% of ThO_2, the average deviation is 0·68% and the time taken for a determination is ≃2 min. K. SAITO

1705. **Determination of ammonium salts in certain substances susceptible of being decomposed to give ammonia on boiling with fixed alkalis.** G. Bourjol (Serv. des Poudres, Poudrerie Nationale, Toulouse, France). *Chim. Anal.*, 1959, **41** (7), 281–286.—Ammonium salts are determined by reaction with formaldehyde soln. and titration of the liberated acid to a phenolphthalein end-point. *Procedure*—(*i*) *In the presence of guanidine nitrate, cyanoguanidine, nitroguanidine, urea, sodium azide or hexamine*—The sample (10 g) is dissolved in 50 ml of water and filtered, and the neutrality is checked with litmus paper. The filtrate and washings are treated with 30 ml of formaldehyde soln. freshly distilled over $CaCO_3$ or $BaCO_3$, and titrated with 0·1 *N* NaOH in the presence of a few drops of 2% phenolphthalein or thymol blue to a barely perceptible colour. After 10 min. at room temp. the titration is continued to a permanent colour. If the original soln. is not neutral, the free acid or alkali is determined by the titration of another sample, with methyl red - methylene blue as indicator, or the free acid is titrated in the same sample to a phenolphthalein end-point before the addition of formaldehyde, and the correction of Ronchèse (*"Guide pratique pour l'Analyse des Urines"*) is applied (1 ml of 0·1 *N* NaOH ≡ 0·00176 g of NH_3 instead of the theoretical 0·00170 g). (*ii*) *In the presence of hydrazine, semicarbazide and their salts*—The sample (5 g) is dissolved in warm water and shaken with 10 ml of benzaldehyde for 5 min. The pptd. hydrazone is dissolved in diethyl ether and the aq. soln. is neutralised to phenolphthalein. Formaldehyde soln. (20 to 25 ml) is added and the soln. is titrated with 0·1 *N* NaOH to a phenolphthalein end-point. With semicarbazide salts alone the addition of benzaldehyde is unnecessary. (*iii*) *In the presence of hydroxylamine salts*—The sample (≃ 10 g) is dissolved and made up to 500 ml. To a 25-ml aliquot is added 20 ml of neutral formaldehyde soln. and the soln. is titrated with 0·5 *N* NaOH to a phenolphthalein end-point (total acidity). The acidity other than that due to ammonium salts is determined in another 25-ml aliquot by adding acetone and titrating to a bromophenol blue end-point. If necessary the acidity of the formaldehyde may be determined separately. E. J. H. BIRCH

1706. **Gasometric determination of ammonia in ammonium salts.** J. Polčin (Res. Inst. Cellulose Ind., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1959, **13** (7–8), 446–453.—The method is based on the oxidation of NH_4^+ with alkaline hypobromite to elementary N, the vol. of which is measured in a simple apparatus (illustrated). Reducing compounds do not interfere. *Procedure*—To a 50-ml flask add $Na_2B_4O_7$ (1·5 to 2 g), KBr soln. (15%) (10 ml) and, with magnetic stirring, a suspension of chlorinated lime (0·35 to 0·40 *N*) (20 ml). After 0·5 to 1 min. stop the stirring and carefully add $CaCl_2$ soln. (15%) (10 ml), H_2O (5 ml) and the sample soln. (≯5 ml, containing 2 to 10 mg of NH_3) so as to avoid mixing the layers. Close the flask with a rubber stopper connected with the measuring apparatus, stir, and measure the vol. of the liberated N. A nomogram is given. J. ZÝKA

1707. **Determination of nitrate ions in mixed acids and nitric esters by electrometric procedures.** W. C. Easterbrook (I.C.I. Ltd., Stevenston, Ayrshire, Scotland). *J. Appl. Chem.*, 1959, **9** (8), 410–417.—The end-point in the Bowman and Scott titration of NO_3^- with $(NH_4)_2SO_4.FeSO_4$ can be determined electrometrically, as a sharp fall in potential of 150 to 250 mV, with bimetallic electrodes (platinum/chromium - nickel) in circuit with a potentiometer or pH meter. An electronically operated traffic-light end-point indicator is used. For mixed acids there is a small but systematic positive error in comparison with the visual method (12 analyses). The electrometric procedure can be extended to nitrocellulose (with different contents of N), nitroglycerin and gun-cotton, after dispersion of the sample in glacial acetic acid and hydrolysis with 80% H_2SO_4. The results agree well with those obtained by the alkaline saponification and Lunge nitrometer methods. W. J. BAKER

1708. **Spectrophotometric determination of phosphorus by the extraction of molybdovanadophosphate with *iso*butyl methyl ketone.** Hiroshi Kitagawa and Norio Shibata (Hitachi Central Res. Lab., Kokubunji, Tokyo). *Japan Analyst*, 1959, **8** (5), 302–306.—For the extraction of molybdovanadophosphate, *iso*butyl methyl ketone (**I**) is the best solvent and the optimum concn. of $HClO_4$ in the aq. layer is 1·2 to 1·6 *N*. A small change in the amount of Mo and V does not affect the extinction coeff. There is no interference from Zn, Mn, Ni, Co, Al, Cu, Mg or Fe (<1 g each for <2 mg of P per 100 ml of the aq. soln.), Cr^{III} (<0·1 g) or As (<0·5 mg). Silicon is removed as SiO_2, and Ti (<50 mg), Sb and Sn (<10 mg each) are masked with citric acid. *Procedure*—Dissolve the sample (0·2 to 0·5 g) in 10 ml of a mixture of HNO_3 and HCl (1 : 1, v/v), add H_2O_2 (3%) (1 ml), reduce the vol. to 5 ml by evaporation,

then add water (50 ml) and $HClO_4$ (10 ml). Cool, add NH_4VO_3 soln. (2·4 g per litre containing 20 ml of 8 *N* HNO_3) (10 ml) and $(NH_4)_2MoO_4$ soln. (150 g of commercial salt per litre) (15 ml), and after 7 min. add citric acid (50%) (10 ml) and extract with **I** (2 × 10 ml). Dry the **I** phase with Na_2SO_4, dilute to 25 ml and measure the extinction at 400 mμ (for < 0·2 mg of P) or at 490 mμ (for < 2 mg of P).

K. Saito

1709. **Carrier-free separation of phosphorus-32 by solvent extraction.** Tsunenobu Shigematsu and Masayuki Tabushi (Inst. for Chem. Res., Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1959, **8** (4), 261–263.—Radioactive H_3PO_4 (containing ^{32}P produced by the irradiation of S with slow neutrons) in 0·5 *N* HCl is extracted with butyl acetate (half the vol. of the aq. phase) in the presence of 0·5% (w/v) of Na_2MoO_4. The ^{32}P is back-extracted with water, the soln. is made 6 to 8 *N* with respect to HCl and the Mo is extracted (×3) with an equal vol. of butyl acetate; carrier-free ^{32}P remains in the aq. phase.

K. Saito

1710. **Colorimetric determination of phosphate with ascorbic acid as reducing agent.** L. Redinger and J. Schmidt (Inst. Org. Chem., Dtsch. Akad. Wissenschaft., Berlin-Adlershof). *Chem. Tech., Berlin*, 1959, **11** (6), 324–326.—In the procedure of Erdey *et al.* (*cf. Anal. Abstr.*, 1955, **2**, 1495) Beer's law is obeyed at 720 or 820 mμ when suitable relative concn., reaction time and temp. are used. A soln. of the sample, with 10 ml of 5 *N* H_2SO_4, 10 ml of 3·5% $(NH_4)_6Mo_7O_{24}.4H_2O$ soln. and 20 ml of 0·1 *N* ascorbic acid, is diluted to 100 ml and set aside for 1 hr. at 20° (thermostat); the extinction is then measured in a Pulfrich photometer with filter S72. Tests on organic phosphates and thiophosphates showed that organic matter could be destroyed by H_2SO_4 - HNO_3 or $HClO_4$, the residual acid being neutralised when making up the soln. for test. The amount of sample must be chosen so as to give 0·1 to 4 μg of P per ml of the blue solution.

A. R. Pearson

1711. **The colorimetric determination of inorganic orthophosphate. I. Determination of inorganic orthophosphate in the presence of some acid-labile phosphate compounds of biochemical significance.** Kenji Mori and Michinori Nakamura (Dept. of Agric. Chem., Univ. of Tokyo, Bunkyo-ku). *Bull. Agric. Chem. Soc. Japan*, 1959, **23** (4), 272–278 (in English).—The determination of inorganic orthophosphate by the colorimetric method of Allen (*Biochem. J.*, 1940, **34**, 858) and its modifications is liable to interference in the presence of acid-labile phosphate compounds [*e.g.*, glucose 1-phosphate, adenosine phosphates, and phosphoryl(enol)-pyruvate] owing to their hydrolysis under acid conditions to liberate inorganic orthophosphate. The effects on this hydrolysis of the amount of Amidol (2:4-diaminophenol hydrochloride) reagent (**I**) and of the temp. at which the molybdenum blue colour is developed have been studied. It is recommended that the determination be carried out at 18° to 20°, at which temp. the rate of colour development is sufficiently rapid for practical purposes (5 to 10 min.) and the developed colour is relatively stable, whilst interference from acid-labile phosphate compounds is negligible. The amount of **I** has less effect, but it should be reduced to one-half of that given in the original method.

II. Determination of total phosphorus. Kenji Mori and Michinori Nakamura. *Ibid.*, 1959, **23** (4), 278–280.—It is shown by colorimetric and radiochemical methods that, during the acid digestion with H_2SO_4 or $HClO_4$ necessary to convert the organic phosphorus compounds into inorganic orthophosphate, the condensation of orthophosphates to polyphosphates, which do not develop a molybdenum-blue colour in the usual methods and would introduce an error into the determination, is negligible. No loss of PO_4^{3-} was observed if fumes were not allowed to escape from the flask during the digestion.

R. A. Brenan

1712. **Rapid analytical methods for the determination of metals and minerals. IX. Determination of arsenic as molybdenum blue.** P. Povondra and Z. Šulcek (Centr. Geol. Inst., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (7), 2393–2397.—The photometric method is based on the reduction of molybdoarsenic acid in an acid medium with ascorbic acid. The excess of molybdate is masked with tartaric acid. For the separation of minute amounts of As, reduction with $(NH_4)_2SO_4.FeSO_4$ with borate as catalyst, distillation with azeotropic HCl and oxidation of the distillate with $KClO_3$ are recommended.

J. Zýka

1713. **Rapid gravimetric method for the determination of arsenates.** M. Vancea and M. Volușniuc (Inst. de Chim., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1959, **10** (1), 141–144.—The method is based on the pptn. of $BiAsO_4.H_2O$. *Procedure*—The soln. (10 to 30 ml) containing ≃0·1 to 0·2 g of alkaline arsenate is diluted to 100 ml and acidified with 1 ml of conc. HNO_3. To the boiling soln. is added dropwise, with continuous stirring, a 0·5% soln. of $Bi(NO_3)_3$ until the clear supernatant liquid no longer gives a ppt., avoiding excess; 1 or 2 drops of the reagent are added to ensure complete pptn., the liquid is set aside for a short time and the ppt. is then collected in a G4 crucible and washed with 2% HNO_3, ethanol and diethyl ether, dried *in vacuo* and weighed. The errors are ≯0·22%.

H. Sher

1714. **Analyses by the use of fluorotitanic acid and hydrogen peroxide. V. Determination of antimony.** Hisao Fukamauchi, Ryuko Enohara, Mitsuko Uehara, Satoko Terui and Yoshiko Tokimoto (Tokyo College of Pharm., Ueno, Taito-ku, Tokyo). *Japan Analyst*, 1959, **8** (6), 353–355.—A reaction similar to that which occurs in the presence of Sn^{4+} (*cf. Anal. Abstr.*, 1960, **7**, 1696) takes place in the presence of Sb^{V}. A reagent of the same molar ratio in 2·5 *N* HCl is the most satisfactory. The working curve is rectilinear for < 18 mg of Sb per 25 ml. The reaction is rapid at room temp.

K. Saito

1715. **Photometric determination of small amounts of vanadium in various materials by the sodium diphenylaminesulphonate method.** Tsutomu Fukasawa, Yoshikazu Takabayashi and Shizo Hirano (Inst. Techno-anal. Chem., Fac. of Engng, Nagoya Univ., Chikusa-ku, Nagoya). *Japan Analyst*, 1959, **8** (5), 292–298.—This method was applied to the analysis of iron ores, Portland cement, manganese dioxide and intermediates in titanium production. For the analysis of ore (0·1 g), the HCl soln. is treated with Fe^{3+} (20 mg), and the V is co-pptd. with Fe. The ppt. is dissolved in H_2SO_4 (1:4) (10 ml) and mixed with H_2SO_4 (1:1) (36 ml), H_3PO_4 (15 ml), $Fe_2(SO_4)_3$ (=0·1 g of Fe), urea soln. (20%) (5 ml), $FeSO_4$ soln. (0·1 *N*) (3 drops) and $KMnO_4$ soln.

(1·5%) until the soln. becomes light purple; after 2 min., the soln. is treated with $NaNO_2$ soln. (3%) until colourless, then made up to 100 ml after the addition of Na diphenylaminesulphonate soln. (0·05%) (2·0 ml), and the extinction is measured at 562 mμ; a few drops of 0·1 *N* $FeSO_4$ are added and the extinction is measured again; the amount of V is calculated from the difference in extinction. Manganese interferes, but V is separated from it by re-pptn. with $Fe(OH)_3$. Since a large amount of Ti does not vitiate the determination, the method is applicable to materials such as ilmenite and titanium slag. The sample (200 mesh, 0·5 g) is fused with Na_2CO_3 (4 g) and Na_2O_2 (4 g), the melt is dissolved in HCl and the V is pptd. with $Fe(OH)_3$. Carbonaceous substances such as oil, coke and pitch (0·5 g) are fused with Na_2CO_3 (2 g) in a current of air, dissolved in HCl and similarly treated. Titanic chloride is dissolved in HCl and metallic titanium in H_2SO_4 (1:1) (20 ml); the product is treated with H_2O_2, the H_2O_2 is decomposed and the soln. is mixed with Fe^{3+}, which are pptd. with aq. NH_3 soln.

K. SAITO

1716. **Determination of niobium and tantalum in binary alloys and zirconium alloys.** D. F. Wood and I. R. Scholes (Res. Dept., I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Anal. Chim. Acta*, 1959, **21** (2), 121–135.—In alkaline soln. pyrogallol reacts with Nb to give a yellow complex much more intensely coloured than the corresponding Ta complex, while in acid soln. the reverse is the case. A simple photometric procedure based on this fact is proposed for alloys containing from 2 to 7% of either Nb or Ta. Differential photometry may be used for samples containing from 7 to 20%. Corrections must be made for the Fe content (≯0·5%), Mo is separated before the determination, and Ti must be absent. *Procedure*—For Nb contents of 2 to 7%, take sufficient sample to contain ≃5 mg of Nb and fuse with $KHSO_4$ (5 g) in a small dry conical flask until dissolved. Cool, extract with a hot soln. of ammonium oxalate (8·0 g in 60 ml), cool, transfer to a 250-ml flask, dilute to volume, mix and transfer 25 ml to a 50-ml flask. Add alkaline pyrogallol soln. (2% in a 20% soln. of $Na_2SO_3.7H_2O$) (20 ml), dilute to volume, mix, and record the extinction after 15 min. at 450 mμ in 2-cm cells. Apply corrections for the Ta or Zr present. For samples containing Ta (2 to 7%) the procedure is the same except that 5 ml of acid pyrogallol soln. (15% in *N* H_2SO_4) is used and the extinction is measured at 400 mμ. Corrections are applied for the Nb or Zr present.

T. R. ANDREW

1717. **Internal electrolysis - coulometric method for determination of small quantities of oxygen. Application to individual samples.** W. G. Knapp (Argonne National Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1463–1467.—The method described is based on the reaction between O and cuprous ammonium chloride and the electroreduction of the Cu^{2+} formed to Cu^+. The current generated is a measure of the O absorbed by the reagent, and is integrated with time by conventional coulometric procedures. The method possesses high sensitivity and good linearity over a wide range of oxygen concn. The sample vol. required is small and the sample can be recovered. The method has been applied to studies in radiation chemistry, reactor water, steam and gas, and to corrosion research.

K. A. PROCTOR

1718. **Separation and determination of microgram amounts of sulphur.** R. P. Larsen, L. E. Ross and N. M. Ingber (Chem. Engng Div., Argonne Nat. Lab., Lemont, Ill.). *Anal. Chem.*, 1959, **31** (9), 1596–1597.—Microgram amounts of S are determined spectrophotometrically at 560 mμ with pararosaniline and formaldehyde after absorption as SO_2 in sodium tetrachloromercurate soln. The S is volatilised as SO_2 by fusion of the sample with V_2O_5 in a combustion tube at 900° to 950°. Copper turnings, also packed into the combustion tube, effect the reduction from SO_3 to SO_2, and at the same time remove any NO_2. The procedure is suitable for determining S in uranium trioxide, sodium zirconium fluoride and hydrofluoric acid.

J. H. WATON

1719. **Rapid spectrophotometric determination of sulphur and sulphate in pyrites, slag and sulphate compounds by the ferric sulphate complex method.** Shigeo Wakamatsu (Toto Seiko Co., Minamisunamachi, Koto-ku, Tokyo). *Japan Analyst*, 1959, **8** (6), 372–377.—The absorption of the Fe^{3+} - SO_4^{2-} complex in $HClO_4$ at 380 mμ was applied to the determination of S. The extinction increases with increase in amount of Fe and $HClO_4$ and with rise of temp. (0·8% per 1° near 25°). The working curve is linear for <60 mg of S per 50 ml of aq. soln. containing 30 ml of $HClO_4$ and 0·3 g of Fe. Interference results from NO_3^-, Cl^-, VO_4^{3-} and MoO_4^{2-}; Cr^{VI} is reduced with H_2O_2. Sulphates are dissolved in hot water (0·1 g per 10 ml) and the NO_3^- and Cl^- are removed by heating to 200° with $HClO_4$ (3 ml) for 3 min. No loss of SO_4^{2-} takes place within 5 min. at <250°. Pyrites and slag (1 g) are fused with Na_2O_2 at ≃800° for 5 min., the Fe is removed as $Fe(OH)_3$ and an aliquot of the filtrate is mixed with the reagent soln. in $HClO_4$. For the preparation of the reagent, iron (5 g) is dissolved in HCl (30 ml), boiled for 10 min. (to remove H_2S), heated to white fumes with $HClO_4$ (30 ml), dissolved in water (20 ml) and made up to 500 ml with conc. $HClO_4$.

K. SAITO

1720. **Co-precipitation in quantitative analysis. Study of the growth of barium sulphate crystals.** A. K. Babko and M. I. Shtokalo (Inst. of Gen. and Inorg. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1959, **25** (7), 779–782.—The growth and ageing of crystals of $BaSO_4$ of different forms, and the co-pptn. of Cl^- and MnO_4^-, are studied by means of an electron microscope. The crystal form tends to be changed by co-pptn., and the amount of co-pptn. is greater with cruciform crystals than with others.

G. S. SMITH

1721. **Interfering electrode reaction during polarography of sulphur dioxide solutions containing oxygen.** C. F. Timberlake (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1959, **84**, 570.—The polarographic reduction products of O, *viz*, H_2O_2 and OH^-, interfere with many polarographic determinations. During polarography of SO_2 soln. it is usual to remove O merely to prevent overlapping of the reduction waves of O and SO_2. Experiments with fruit juices and ciders containing citrate - NaOH buffers and SO_2 showed that, although well-defined reduction waves for SO_2 were obtained, their heights depended on the amounts of dissolved O present. It seems probable that the low heights of reduction waves of SO_2 observed during polarography of SO_2 soln. containing O are caused by a reaction at the diffusion layer between SO_2 and H_2O_2 formed by the preceding reduction of O.

A. O. JONES

1722. **Analysis of a mixture of ammonium salts of iminodisulphonic, sulphamic, nitrilotrisulphonic and sulphuric acids.** K. Lasiewicz and B. Byczyńska (Inst. of Sulphuric Acid and Phosphate Fertilisers, Luboń, Poland). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 1027–1031 (in Polish).—A scheme is presented involving the following determinations—(*a*) total ammonium N by distillation, (*b*) total N by Kjeldahl, (*c*) sulphamic acid by titration with $NaNO_2$, with the Griess - Ilosvay reagent as indicator, (*d*) sulphate as $(NH_4)_2SO_4$, as $BaSO_4$, or with benzidine chloride, (*e*) sulphate after complete hydrolysis with HCl in the presence of $NaNO_2$, (*f*) pptn. of iminodisulphonic and nitrilotrisulphonic acids by adding 0·5 *N* KOH and setting aside for 24 hr. followed by determination of SO_4^{2-} in the filtrate. From these results the composition of the mixture can be calculated.

W. B. Miaskowski

1723. **Use of ferric hydroxide for separating quadri- and sexa-valent selenium.** V. I. Plotnikov (All-Union Mining Metallurgy Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (6), 666–668.—Co-precipitation of Se^{IV} with $Fe(OH)_3$ serves to separate Se^{IV} and Se^{VI}. Re-precipitation is advisable.

G. S. Smith

1724. **Polarographic determination of tellurium in copper alloys.** I. E. Bŭkov and L. S. Gorshkova (Inst. of Metallurgy, Ural Filial of Acad. Sci., USSR). *Zavod. Lab.*, 1959, **25** (6), 674–676.—The sample is dissolved in dil. HNO_3, the soln. is evaporated after the addition of HCl, and the residue, after being moistened with water or HCl, is mixed with an electrolyte containing KCN (0·65 *M*), NaOH (0·25 *M*), Na_2SO_3 (0·2 *M*) and gelatin (0·02%). The polarographic wave due to Te at $\simeq -1{\cdot}2$ V is measured. Its height is proportional to the concn. The interference of Cd and Pb can be prevented by the use of EDTA. It is then preferable to omit the use of gelatin and to measure the height of the maximum.

G. S. Smith

1725. **Highly accurate and precise method for the assay of chromium metal and chromium(III) oxide.** H. J. Keily, A. Eldridge and J. O. Hibbits (Gen. Engng Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (2), 135–140.—The sample (0·5 g) is dissolved in acid [30 ml of H_2SO_4 (1 : 1) for chromium metal; 20 ml of H_2SO_4 - $HClO_4$ (1 : 1) for Cr_2O_3] and diluted to $\simeq$250 ml with water. Chromium is oxidised to Cr^{VI} by the addition of solid $(NH_4)_2S_2O_8$ to the boiling soln. containing HNO_3 (1 : 1) (6 ml) and $AgNO_3$ soln. (5%) (10 ml) and the liquid is cooled. A 50-mg excess (assuming 100% purity of the sample) of $(NH_4)_2SO_4.FeSO_4.6H_2O$ is added and the excess is titrated with 0·02 *N* $K_2Cr_2O_7$. Results show standard deviations of $\pm$0·028 (chromium metal, 12 determinations), $\pm$0·055 (Cr_2O_3, sample A, 4 determinations) and $\pm$0·13 (Cr_2O_3, sample B, 4 determinations).

T. R. Andrew

1726. **Determination of chromium in products of the cement industry.** R. Flatt and P. Cusani (Lab. de Chim. Minérale et Anal., Univ. de Lausanne, Switzerland). *Anal. Chim. Acta*, 1959, **21** (2), 181–186 (in French).—Total Cr is determined in the powdered sample (cement or clinker) by extracting the product of fusion with sodium borate into H_2SO_4, adding aq. NH_3 to precipitate Fe and Al, filtering off the ppt. and oxidising the Cr in the filtrate, after acidification, by boiling with $K_2S_2O_8$ in the presence of $AgNO_3$. Diphenylcarbazide is used as the final colour-forming reagent. 'Soluble' chromate is determined on an aliquot of the extract of 20 g of the finely powdered sample with 500 ml of cold water, by the diphenylcarbazide method. Results are presented for samples containing from 13 to 250 p.p.m. of total Cr and from 1 to 20 p.p.m. of soluble Cr^{VI}.

T. R. Andrew

1727. **Analysis of high-chromium slag by acid decomposition method.** Ohiko Kammori and Akira Amano (Res. Lab., Yawata Iron and Steel Works, Yawata, Fukuoka-ken). *Japan Analyst*, 1959, **8** (5), 319–320.—The loss of other ingredients in high-chromium steel (>5% of Cr) on the removal of Cr with $HClO_4$ and HCl is negligible. The sample (0·3 g) is heated with $HClO_4$ (60%) (25 ml) on a hot-plate at 200° until white fumes appear, then treated with HCl (10 *N*) (5 × 5 ml). The Si, Mn, Ca, Mg, Al and Fe are determined by the usual method.

K. Saito

1728. **Complexometric titration of molybdenum. II.** E. Lassner and R. Scharf (Metallwerke Plansee A.-G., Reutte/Tyrol, Austria). *Z. anal. Chem.*, 1959, **168** (6), 429–433 (in German).—A modification of an earlier method (*cf. Anal. Abstr.*, 1960, **7**, 107) enables milligram amounts of Mo to be determined. An almost neutral soln. containing 0·5 to 5 mg of Mo is treated with an excess of 0·005 *M* EDTA, about 3 g of tartaric acid, 3 g of hydrazine sulphate and 2·5 ml of H_2SO_4 (1 : 1), and the mixture is diluted to 100 to 150 ml with water and boiled for 5 min. The soln. is then brought to pH 4·5 to 5·0 with aq. NH_3, 4 drops of 1% soln. of 1-(2-pyridylazo)-2-naphthol indicator are added and the mixture is titrated with 0·005 *M* copper soln. to a deep-red colour and with EDTA soln. to a pure yellow. Typical results are tabulated. The error is $\pm$0·02 mg of Mo. Chloride, sulphate, nitrate, phosphate, acetate, tartrate, citrate, alkali metals, alkaline-earth metals and Mn^{2+} do not interfere. The effect of Ti^{4+}, Nb^{5+}, Ta^{5+}, W^{6+}, Th^{4+}, Al^{3+}, Ce^{3+}, La^{3+} and U^{4+} can be masked by the addition of tartaric acid and fluoride; Bi^{3+}, Cd^{2+}, Zn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Hg^{2+}, V^{4+}, Cr^{3+} and Pb^{2+} interfere by complex formation with EDTA, and Fe^{3+} by reduction of Cu. Procedures for the analysis of Mo - W and Mo - Ta alloys are outlined. The relative maximum error is $\pm$0·5%.

S. M. Marsh

1729. **The micro-identification and the colorimetric micro-determination of molybdenum(VI) with morin.** G. Almássy and M. Vígvári (Forschungsinst. f. Phys. Ungarischen Akad. Wissenschaften, Debrecen, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (3), 243–251 (in German).—Identification is carried out by adding to the sample soln. (5 ml, 0·1 to 0·5 *N* in HCl) 3 ml of 2·5% EDTA soln. and 0·5 ml of satd. NaF soln. After the addition of 2 ml of *n*-butanol and 0·5 ml of 0·33% morin soln., the tube is shaken for 10 to 15 sec. If Mo is present the *n*-butanol phase shows an orange - yellow colour, but if not the *n*-butanol remains colourless. As little as 0·05 μg of Mo in 5 ml of soln. can be detected. *Procedure for determination*—The sample soln. (20 ml containing 20 μg of Mo) is acidified with 0·5 ml of HCl (1 : 1) and extracted with 5 ml of *n*-butanol and 0·5 ml of 0·33% morin soln. in *n*-butanol. The aq. layer is then separated and extracted again twice, each time with 3 ml of *n*-butanol and 0·5 ml of reagent.

The combined extracts are made up to 15 ml and the extinction is measured at 430 mμ against extracts obtained similarly from a Mo-free soln. The effects of pH and morin concn. on the extraction are discussed and the construction of a calibration curve is described. Interfering ions are listed, but their effect can be overcome by the addition of EDTA and NaF. S. M. MARSH

1730. **New volumetric method of determining uranium.** V. G. Goryushina and T. A. Archakova (State Sci. Res. Inst. of Rare Metals). *Zavod. Lab.*, 1959, **25** (7), 789–790.—The weakly acid (HCl) soln. of U is treated with 15% EDTA soln. (disodium salt) (2 to 4 ml in excess of that required to combine with interfering elements) and 3 ml of 30% H_2O_2 soln. if Ti is present and 1 ml of HBF_4 if Zr is present. Then 10 ml of 20% Na_2HAsO_4 soln. and 15 ml of acetic acid are added and the soln. is treated with 50% ammonium acetate soln. to give a violet colour (pH ≃3) with Congo red indicator. The soln. (≯100 ml) is heated on a boiling-water bath to coagulate the ppt., which is filtered off from the cooled soln. and washed with 1% NH_4NO_3 soln. The ppt. is dissolved in 60 to 70 ml of dil. HCl (1:3), the soln. is treated with 10 ml of 30% KI soln., and the iodine liberated after 30 min. is determined in the diluted soln. (150 ml) by titration with 0·025 *N* $Na_2S_2O_3$. G. S. SMITH

1731. **Micro-determination, selective separation and concentration of uranium, using Schiff's bases.** E. Bayer and H. Möllinger (Inst. f. Organ. Chem., Karlsruhe, W. Germany). *Angew. Chem.*, 1959, **71** (13), 426–428.—Uranium forms organic complexes as stable as the corresponding ones of Ni and Co, and in the method described bis-(2-hydroxyphenylimino)glyoxal is used as the complexing agent. Cobalt, Cu, Ni, Zn, Cd, Mn and U all behave similarly, but the complex of U is soluble in water, $CHCl_3$ and the alcohols, whereas the complexes formed by the other metals are only sparingly soluble in water or organic solvents (except pyridine). The treatment of an aq. soln. of the crude uranium salts (4 ml, 10 μg per ml) with a methanolic soln. of the reagent (3 ml, 100 mg per 100 ml, dissolved at 50° and filtered at room temp.) yields a coloured soln. with an absorption peak at 565 mμ, which is specific for U. The colour is developed in 10 min. at 80° to 95° and is stable for 3 hr. A measured aliquot of the soln. is diluted with methanol. The depth of colour reaches a steady maximum when the methanol to water ratio is >1:1 and the pH lies between 3·4 and 5·0 (acetate buffer, pH 4·46). On the linear curve shown a uranium content of 18 μg per ml corresponds to an extinction of 1·0. Details are also given of the concentration of uranium by making use of the poly(glyoxaltriaminophenol) complex, from which the metal is regenerated by the addition of acid. E. G. CUMMINS

1732. **Thermometric titration of uranium(IV) with potassium dichromate.** F. J. Miller and P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (2), 112–116.—The use of a platinum indicator electrode in the potentiometric titration of U^{IV} with $K_2Cr_2O_7$ is not satisfactory. It is shown that good results (standard error ±1% rel.) are obtained by using the automatic thermometric procedure of Linde *et al.* (*Anal. Chem.*, 1953, **25**, 404). T. R. ANDREW

1733. **Spectrophotometric determination of uranium with acetylacetone.** Tsunenobu Shigematsu and Masayuki Tabushi (Inst. Chem. Res., Kyoto Univ., Sakyo-ku). *Japan Analyst*, 1959, **8** (4), 253–256.—Krishen's method (*Anal. Chem.*, 1957, **29**, 288) was modified to enable the extinction (at 350 to 370 mμ) to be measured in an aq. soln. The extinction is dependent on the pH, reaching a max. at pH 6·5 to 7·5. The composition of the U - acetylacetonate appears to differ from that extracted into an org. layer. Interference results from Bi, Cr, Cu, Fe, Pb and Ti (these form ppt.), and from Mn, V and W (these give a yellow coloration). The interference of EDTA, which masks Bi and Pb, is eliminated in the presence of Ca. K. SAITO

1734. **The application of square-wave polarography to the monitoring of the uranium content of uranium plant solutions.** E. Goldblatt (Stilfontein Gold Mining Co. Ltd.). *J. S. Afr. Inst. Min. Metall.*, 1959, **59** (7), 369–382.—The accuracy of square-wave polarography for determining the U content of soln. has been shown to vary from a coeff. of variation of 2·8% for a concn. of 0·3 g of U_3O_8 per litre to 5·4% for a concn. of 0·01 g of U_3O_8 per litre. The method is sufficiently simple to be used for the continuous monitoring of plant soln. K. A. PROCTOR

1735. **Radiometric analysis of naturally leached uranium - thorium ore samples: comparison of different methods.** P. W. de Lange (Radioactivity Div., Nat. Phys. Res. Lab., Pretoria, S. Africa). *J. S. Afr. Min. Metall.*, 1959, **59** (12), 640–656.—The results obtained with the "β-γ," the "γ-discrimination" and the "β-γ-γ" methods are compared. The "β-γ-γ" method does not rely, as does, *e.g.*, the method of Eicholz *et al.* (*Canad. J. Phys.*, 1953, **31**, 613) on specific ratios of the calibration constants for the determination of ThO_2, but is a combination of the "β-γ" and the "γ-discrimination" methods which enables U_3O_8 and ThO_2, and the equilibrium values of the U series, to be obtained. Drift-free apparatus is necessary for the routine application of the "β-γ-γ" method, but commercially available equipment can be used, with slight modification. The min. concn. that can be determined are 0·005% of U_3O_8 and 0·005% of ThO_2 in mixed naturally leached ore with 90-g samples. J. M. JACOBS

1736. **Effect of thorium on the determination of uranium with morin.** S. Orsós. *Nehézvegyipari Kutató Intézet Közleményei*, 1959, **1** (3–4), 321–324.—Provided that its concn. is ≯100 mg per 25 ml, Th does not interfere in the colorimetric determination of U with morin at 470 mμ (Pulfrich photometer with monochromator VM-2). Higher concn. of Th should be removed by an initial extraction with HNO_3 - diethyl ether. (From English summary.) W. J. BAKER

1737. **The use of organic reagents in the polarographic determination of uranium and zirconium.** Masayoshi Ishibashi, Taitiro Fujinaga and Kosuke Izutsu (Inst. of Chem., Univ. of Kyoto, Japan). *J. Electroanal. Chem.*, 1959, **1** (1), 26–38.—Systematic investigation of the polarographic behaviour of *oo'*-dihydroxyazo and *o'*-amino-*o*-hydroxyazo dyes has led to the selection of Mordant Blue 2R (C.I. Mordant Blue 9) (**I**), as a suitable reagent for the determination of U and Zr. **I** and UO_2^{2+} in an acetate (2·0 *M*) buffered soln. at pH 5·3 produce well-defined combined waves in the polarogram,

and the height of the second wave, which appears at a potential more negative than that of the dye alone, is proportional to the concn. of UO_2^{2+}. Similar results are obtained for ZrO^{2+} at a pH of about 1·5, unbuffered. The removal of interfering ions is described. E. G. CUMMINS

1738. **Polarographic analysis of uranium. Separation of concomitant ions with anion-exchange resin.** Mutsuaki Shinagawa, Toshifumi Murata and Hiroshi Okashita (Fac. of Sci., Hiroshima Univ., Higashi-senda-machi). *Japan Analyst*, 1959, **8** (6), 356–361.—The separation of UO_2^{2+} from Ti^{IV}, Ni, Cu^{II}, Sn^{IV}, Bi, Sb, Cr, Co, Cd, As, MnO_4^-, VO_4^{3-} ($< 10^{-3}$ *M*) and Fe^{III} ($< 4 \times 10^{-4}$ *M*), which interfere with the polarographic determination of U in 0·5 *N* HCl, is effected by the use of Amberlite IRA-400 (sulphate form) (50 mesh). The sample, in 0·1 to 0·2 *N* H_2SO_4 soln., is passed (1 drop per sec.) through a column (diam. 1·2 cm, length 7 cm) of the resin, which is washed with water (100 ml) and eluted with *N* $HClO_4$ (50 ml). (Hydrochloric acid is a less satisfactory eluent.) The eluate is diluted to 100 ml with water containing 2 ml of 0·1% Tween 80 and the polarogram is recorded ($E_{\frac{1}{2}}$ 0·51 and −1·43 V *vs.* the S.C.E.). The height of the second wave is proportional to the concn. of U ($< 10^{-4}$ *M*). In the presence of MoO_4^{2-} ($< 3 \times 10^{-4}$ *M*), the H_2SO_4 soln. is heated with hydroxyammonium sulphate (0·5 g). The method can be applied to uranium ores. K. SAITO

1739. **Determination of a micro amount of impurities in metallic uranium. IV. Photometric determination of copper.** Masami Suzuki (Inst. Techno-anal. Chem., Fac. of Engng, Nagoya Univ., Chikusa-ku). *Japan Analyst*, 1959, **8** (6), 395–397.—The diethyldithiocarbamate (**I**) method for Cu (*cf.* Chilton, *Anal. Abstr.*, 1954, **1**, 24) was modified to mask interfering ions such as Ni and Co with EDTA and to extract the Cu - **I** complex with butyl acetate (**II**). Since EDTA retards the extraction, the amount of **I** and the shaking time must be increased. *Procedure*—Dissolve the sample (< 1 g, containing 1 to 10 μg of Cu) in HNO_3 (3 ml), expel the excess of acid, dissolve the residue in water, add citric acid soln. (25%, w/v) (10 ml), adjust to pH 9 with aq. NH_3 soln., add EDTA soln. (4%) (1 ml), dilute to 25 ml, add **I** soln. (sodium salt) (1%) (1·0 ml) and shake with **II** (5·0 ml) for 3 min. Wash the **II** phase with H_2SO_4 (2 *N*) (5 ml) by shaking for 30 sec., and measure the extinction at 440 mμ. K. SAITO

1740. **Determination of nitrogen in uranium nitrides.** J. Lathouse, F. E. Huber, jun., and D. L. Chase (Battelle Memorial Inst., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1606–1607.—A modified Kjeldahl technique is used for the determination of N in both UN and U_2N_3. The sample is digested with HCl (1 : 1) in the presence of HF and $CuSeO_4$. Acceptable results are obtained for both types of nitride. J. H. WATON

1741. **Distribution of neptunium between tri-*n*-butyl phosphate and some mineral acids.** Tomitaro Ishimori and Eiko Nakamura (Chem. Div., Japan Atomic Energy Res. Inst., Tokai, Ibaraki). *Bull. Chem. Soc. Japan*, 1959, **32** (7), 713–720 (in English).—Neptunium-239 is used as the tracer for studies on solvent-extraction behaviour of Np in the systems of tri-*n*-butyl phosphate with HNO_3, HCl, H_2SO_4 or $HClO_4$ soln. Partition coeff. are measured by combinations of scrubbing techniques and chemical treatments. A simple method for the isolation of neptunium from irradiated uranyl salt is described. I. JONES

1742. **Determination of chloride ion by conductimetric titration with mercuric nitrate solution.** G. B. Pasovskaya (Turkmenian Med. Inst., Askhabad, USSR). *Lab. Delo*, 1959, **5** (4), 19–21.—*Procedure*—With a micro-pipette, measure out the sample (natural water) (5 to 10 ml), dilute to 25 ml, add methyl orange (1 drop) and *N* HNO_3 (2 to 3 drops in excess after neutralisation) and titrate conductimetrically with 0·1 *N* mercuric nitrate. The method can be used to determine down to 0·22 mg of Cl^- in 25 ml of soln. With smaller amounts of Cl^- the error increases. The presence of > 25-fold amounts of K^+, Na^+, NH_4^+, Mg^{2+}, Ca^{2+}, Al^{3+}, Fe, NO_3^-, CO_3^{2-}, HCO_3^-, silicate and SO_4^{2-} does not cause interference. To eliminate the interference of > 380 mg of SO_4^{2-}, add 6 to 7 drops of *N* HNO_3 after neutralisation. K. R. C.

1743. **Coulometric titration of manganese with electrogenerated vanadyl ion.** D. G. Davis (Georgia Inst. of Technol., Atlanta, Ga., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1460–1463.—The manganous ion can be converted into MnO_4^- with silver oxide before titration in H_2SO_4 with electrogenerated vanadyl ion. The titration is specific. Chromium interferes, but this interference can be considerably reduced by the correct choice of conditions. K. A. PROCTOR

1744. **Rapid analytical methods for the determination of metals and minerals. X. Use of ion exchange for the determination of manganese and alkaline earths.** P. Povondra and Z. Šulcek (Centr. Geol. Inst., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (7), 2398–2404.—Manganese and alkaline-earth metals can be separated by adsorption on Amberlite IR-120. Manganese, Mg and alkaline-earth metals adsorbed on the column can be eluted with a standard soln. of EDTA (disodium salt) (**I**) by varying the acidity. *Procedure*—Carbonates are decomposed with HCl, then oxidised with HNO_3, and after repeated evaporation with HCl the undissolved residue is filtered off. Silicates with a high Mn content are given a preliminary treatment with acids. Difficultly soluble silicates are evaporated with HF and H_2SO_4 and the residue is dissolved in HCl. Tungsten-containing materials are dissolved by treatment with conc. HCl; tungstic acid is separated after treatment with HNO_3, the excess of which is removed by evaporation with HCl. *Separation*—Evaporate an aliquot of the dissolved sample to dryness on a sand bath; to the residue add citric acid (3 g) and dilute to 100 ml. Adjust the pH to 3·3 to 4 and dilute to 150 ml. Pour the soln. through a column containing Amberlite IR-120 (2 to 3 ml per min.) and wash thrice with 13 ml and once with 50 ml of H_2O. Elute the Mn from the column with acetate buffer soln. (pH 4·6) (15 ml) containing a known vol. of 0·05 M **I**. Use the same procedure for the elution of Ca and Mg with **I** and borate buffer soln. (pH 11). Neutralise the eluates, add aq. NH_3 - NH_4Cl buffer soln. and titrate the excess of **I** with 0·05 *M* $ZnSO_4$. Manganese and the sum of Ca and Mg are so determined. Calcium can be separated from a second aliquot as the oxalate, and determined complexometrically. J. ŽÝKA

1745. **Separation of technetium from rhenium by electrophoresis and paper chromatography.** R. A. Guedes de Carvalho (Oporto Univ., Oporto, Portugal). Thesis, Univ. of Oporto, 1958, 164 pp.—Conditions for the separation of Tc from Re, and in some cases also from Mo, have been studied. Good separations were obtained by electrophoresis when alkaline hydrazine sulphate, $SnCl_2$ - HCl, HBr or HI were used as electrolytes. Satisfactory separations were also obtained by paper chromatography, in conjunction with a reducing treatment, *e.g.*, by using a paper impregnated with hydrazine sulphate at pH 8·95 and eluting with ethanol, or by using butanol saturated with HI as an eluent. The reduction of Re^{7+}, and complexes of Re^{4+}, has also been studied chromatographically. The best reagents for locating Re^{4+} on paper are Alizarin red S (C.I. Mordant Red 3), ammoniacal 8-hydroxyquinoline and dithio-oxamide.

L. A. O'NEILL

1746. **Rapid photometric determination of a small amount of iron in non-ferrous metals by the cupferron extraction method.** Shigeo Wakamatsu (Toto Seiko Co., Minami-sunamachi, Koto-ku, Tokyo). *Japan Analyst*, 1959, **8** (5), 298–302.—The extraction of Fe - cupferron is best effected with *iso*amyl acetate (**I**) from an aq. soln. of pH 1·0 to 3·0 (Beckwith, *Anal. Abstr.*, 1954, **1**, 2415). There is no interference from metals other than V and Ti in the presence of EDTA. The time taken for a determination is ≃20 min. *Procedure*—Dissolve lead (0·2 g) in 3 ml of a mixture of $HClO_4$ (60%) (50 ml) and HNO_3 (10 ml), heat almost to dryness, dissolve the residue in water (10 ml) and add EDTA (disodium salt) soln. (5%) (10 ml) and ammonium acetate soln. (50%) (10 ml). Adjust the pH to 3·0, dilute the soln. to 50 ml, add fresh cupferron soln. (5%) (5 ml), shake with **I** (10 ml) for 1 min. and measure the extinction of the **I** layer at 420 mμ. Dissolve tin (0·2 g) in 5 ml of a mixture of HCl (180 ml) and Br (20 ml), heat to remove Br and add water (10 ml). Dissolve zinc, nickel and manganese in aqua regia (5 ml), heat to white fumes with $HClO_4$ (3 ml), and dissolve the residue in water (10 ml). K. SAITO

1747. **Manganimetry. III. Determination of a small amount of iron in the presence of arsenic, antimony and tin with manganic pyrophosphate.** Masayoshi Ishibashi, Tsunenobu Shigematsu and Shozo Shibata (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1959, **8** (6), 377–379.—By the use of a standard manganic pyrophosphate soln. (*cf. Anal. Abstr.*, 1959, **6**, 3378), Fe^{3+} (0·2 to 2 mg) can be titrated in 0·7 *N* H_2SO_4 in the presence of <6 mg of Sb^{III}, <5 mg of Sn^{II} and <10 mg of As. The presence of tartaric acid (<2%) does not affect the titration. The amount of Sb and Sn tolerated is several times as great as that by the use of Ce^{IV} soln. K. SAITO

1748. **The determination of mixtures of iron and cerium by controlled-potential coulometric analysis with a platinum electrode.** D. G. Davis (Georgia Inst. of Technol., Atlanta, Ga., U.S.A.). *J. Electroanal. Chem.*, 1959, **1** (1), 73–78.—Accurate results for mixtures of Ce and Fe are reported in this account of the reduction of several oxidants at a platinum-gauze electrode. The coulometric reduction of Ce (at +0·80 V *vs.* the S.C.E.) and Fe (at +0·20 V) followed exhaustive electrolytic oxidation. The electrode was changed between these operations. The application of a correction for surface oxidation of the electrode is discussed.

E. G. CUMMINS

1749. **Separation of iron from manganese by means of benzoic acid and hexamine, and the quantitative determination of these metals in the absence of chlorine ions.** R. Ripan and I. Pîrvu (Univ. "V. Babeş", Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1959, **10** (1), 129–135.—The method is based on the pptn. of Fe as benzoate in a boiling soln. buffered with hexamine at a pH between 3 and 5 in the presence of a small amount (0·5 to 1 g) of ammonium salts. *Procedure for the determination of Fe*—A soln. of 0·03 to 0·5 g of Fe and 0·02 to 0·15 g of Mn in ≯1·5 ml of conc. HNO_3 is diluted to ≃100 ml and treated in the cold with 10 ml of a 9% ethanolic soln. of benzoic acid (**I**). The gently boiling soln. is treated dropwise with a 10% aq. soln. of hexamine (**II**) until the mixture is red. Thereafter **II** is added less rapidly, with constant stirring and gentle boiling until the liquid becomes turbid. Boiling and stirring is continued for 2 min., and a further 5 ml of **II** is added dropwise; after a further 5 min., 3 to 5 drops of 3% H_2O_2 soln. are added, and the soln. is re-boiled. The boiling soln. is decanted through a filter-paper after the ppt. has settled, and the ppt. is washed 2 or 3 times by decantation with boiling wash soln. (500 ml of boiling water, to which is added 2 ml of **II** and 8 ml of **I**), setting aside for 1 min. between each washing. The ppt. is then washed on the filter-paper 7 or 8 times with boiling wash soln. (a total of ≃200 ml). The ppt. is dried, ignited gently in a porcelain crucible, and then calcined to Fe_2O_3 at 800° to 900°. The neutralisation of the original test soln. to the appearance of the red colour can be achieved by the addition of a 2% aq. soln. of NH_3 instead of **II**. There is a loss by volatilisation of $FeCl_3$ in the presence of Cl^-, even when large quantities of ammonium salts are present. *Determination of Mn*—The filtrate from the pptn. of Fe (≃300 ml) is treated with 3 or 4 drops of conc. HCl and evaporated to 200 ml. Conc. HCl (5 ml) and a soln. of 1·7 g of $(NH_4)_2HPO_4$ is added to the hot soln., followed by conc. aq. NH_3 until a persistent slight ppt. appears. This is re-dissolved with a few drops of conc. HCl and re-pptd. by the addition of dil. aq. NH_3 to the boiling soln. The ppt. is dried and calcined to $Mn_2P_2O_7$ in the usual manner. The errors are $< \pm 0{\cdot}5\%$ for both Fe and Mn. H. SHER

1750. **Polarography of mixtures. Simultaneous determination of iron and nickel.** S. L. Phillips and E. Morgan (Research Lab., International Business Machines Corp., Poughkeepsie, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1467–1469.—Iron and Ni can be determined with a precision of ±2% by the method described. No preliminary separation is required. Supporting electrolytes of aq. NH_3 - NH_4Cl or pyridine - NH_4Cl containing an excess of 5-sulphosalicylic acid are used. K. A. PROCTOR

1751. **Extractive determination of iron and cobalt in pure nickel.** S. E. Kreĭmer and L. P. Butylkin ("Severonikel'" Combine). *Zavod. Lab.*, 1959, **25** (6), 662–666.—To determine Co by extraction in the presence of diantipyrinylmethane (**I**) a calibration curve is prepared by means of aliquots (1 to 10 ml) of a standard soln. of $CoSO_4$ containing 0·01 mg per ml. Each portion is treated with two drops of conc. HCl, 5 ml of 40% NH_4SCN soln. and 10 ml of a 2% soln. of **I** in 0·5 *N* HCl. The soln. is well

shaken and then extracted with $CHCl_3$ (2 × 5 ml). The extract is then shaken with a buffer soln. of pH 5·37 (acetic acid - Na acetate) (2 × 10 ml) and the combined aq. extracts are boiled for 5 min. after the addition of 1 ml of glacial acetic acid and 5 ml of 50% Na acetate soln. Then 10 ml of 0·1% nitroso-R-salt soln. is added and the soln. is boiled again; 5 ml of dil. HNO_3 (1:1) is added, boiling is continued for 2 min., and the cooled soln. is diluted to 100 ml. A blank is prepared by addition of all the reagents to water, but extraction is not necessary. *Procedure for Co in pure nickel*—The sample (2·5 g) is dissolved in dil. HNO_3 (3:2). The soln. is diluted to 200 ml and an aliquot (20 ml) is evaporated to dryness, followed by the addition of 5 ml of conc. HCl and re-evaporation. The residue is treated with the minimum amount of 0·5 *N* HCl and the soln. is treated as described for the calibration soln. Satisfactory results were obtained with samples containing from 0·0008 to 0·07% of Co. *Procedure for Fe*—The sample (10 g) is dissolved in 100 ml of dil. HNO_3 (3:2) and the soln. is evaporated to low bulk, then diluted to 200 ml. An aliquot (20 or 10 ml) is treated with 5 ml of conc. HCl, evaporated nearly to dryness, treated with 5 ml of conc. HCl and again evaporated. Salts are dissolved, if necessary, in 10 ml of 0·5 *N* HCl, the soln. is mixed with 1 or 2 drops of 0·01 *N* $KMnO_4$ and 1 ml of conc. HCl, and treated with 10 ml of 40% NH_4SCN soln. and 15 ml of a 2% soln. of **I** in 0·5 *N* HCl. After being shaken until a ppt. appears, the soln. is extracted with $CHCl_3$ (2 × 10 ml) and the extract is washed with *N* H_2SO_4 (2 or 3 × 10 ml). The solvent layer is treated with 10 ml of the acetate buffer soln. and 1 ml of 30% hydroxyammonium chloride soln. and shaken. This treatment is repeated on the separated layer, which is then washed with 10 ml of 0·2 *N* H_2SO_4. In the aq. soln. Fe is determined colorimetrically by means of 1:10-phenanthroline. The method is satisfactory for contents >0·004%. G. S. SMITH

1752. **Carbide analysis of white cast iron.** I. E. Lev (Dniepropetrovsk Metallurgical Inst.). *Zavod. Lab.*, 1959, **25** (6), 656–658.—The electrolytic separation of the carbide phase is best carried out at a c.d. of 10 to 15 mA per sq. cm for 3 hr. at room temp. in 0·3 *N* HCl. G. S. SMITH

1753. **Determination of magnesium in nodular cast iron using a chromatographic separation.** K. G. McLaren (Defence Standards Lab., Sydney, Australia). *Anal. Chim. Acta*, 1959, **21** (2), 165–169.—Dissolve the sample (1 g) in HCl (5 *N*) (30 ml), oxidise with HNO_3 (15 *N*) (5 ml), evaporate to dryness and bake for 5 min. at 200°. Dissolve the residue in HCl (10 *N*) (15 ml), add 25 ml of water, heat nearly to boiling and filter. Wash the residue with HCl and hot water, and evaporate the filtrate to dryness. Re-dissolve in HCl (10 *N*) (4 ml) with gentle warming, cool and add 10 ml of freshly prepared elution mixture (98 ml of ethyl methyl ketone and 2 ml of 10 *N* HCl). Swirl and pour on to a cellulose column conditioned by successive washes with 50 ml of HCl (*N*), 150 ml of water, 50 ml of ethyl methyl ketone, and 100 ml of elution mixture. Elute at 1 to 2 ml per min. until 100 ml has passed through the column; the eluate is rejected. The column is then eluted with 80 ml of HCl (1%), 1 g of NH_4Cl is added to the eluate, which is made ammoniacal with 5 *N* aq. NH_3 (2 ml in excess) and heated to boiling. Ammonium oxalate soln. (10%) (2 ml) is added and the ppt. formed is filtered off and washed with water. To the filtrate are added 10 ml of KCN soln. (10%), 5 ml of buffer soln. (6·75 g of NH_4Cl and 57 ml of 15 *N* aq. NH_3 per 100 ml) and ascorbic acid (1 g), and the mixture is diluted to 150 ml. The Mg is titrated with EDTA (disodium salt) (3·7 g per litre), with Eriochrome black T as indicator. The coeff. of variation is $\simeq \pm 10\%$ for contents of Mg of 0·03 to 0·07%. T. R. ANDREW

1754. **Isolation of structural components of iron - aluminium - carbon alloys.** H. Tůma and M. Vyklický (Res. Inst. Materials and Technol., Prague). *Hutn. Listy*, 1959, **14** (8), 706–710.—Electrolytic isolation in a soln. of 5% citric acid and 2·5% NaCl in 0·5 *N* HCl is recommended. A temp. of $\geqslant 3°$ and current density of 0·02 amp. per sq. cm must be maintained. The isolation is complete within 2 hr. For the isolation of aluminium carbide materials, which decompose in contact with water, a procedure is recommended based on the dissolution of the metallic portion of the alloy in an anhyd. soln. of bromine in methyl acetate. J. ZÝKA

1755. **Experience with direct-recording spectrographs in the steel-works laboratory. I.** A. Pack and B. Zischka (Maxhütte A.-G., Sulzbach-Rosenberg, Germany). *Arch. Eisenhüttenw.*, 1959, **30** (7), 407–410.

II. G. Hartleif and H. Kornfeld (Dortmund-Hörder Hüttenunion A.-G., Dortmund-Hörde, Germany). *Ibid.*, 1959, **30** (8), 485–488.

III. P. Dickens and A. Bähr (Mannesmann A.-G., Duisberg-Huckingen, Germany). *Ibid.*, 1959, **30** (8), 489–491.

IV. V. Mathien, M. Lacomble and L. Charlet (S.A. Cockerill-Ougrée, Seraing, Belgium). *Ibid.*, 1959, **30** (8), 493–496.

V. A. Brandt and W. Berstermann (Georgsmarienhutte, Kr. Osnabrück). *Ibid.*, 1959, **30** (9), 541–543.

VI. H. Manecke and W. Brokopf. *Ibid.*, 1959, **30** (9), 545–548.

VII. P. Höller (Hüttenwerk, Oberhausen, Germany). *Ibid.*, 1959, **30** (10), 589–594.

VIII. G. Graue and R. Marotz (Phoenix-Rheinrohr A.-G., Duisburg-Ruhrort, Germany). *Ibid.*, 1959, **30** (10), 595–598.

In these eight papers the authors describe their experiences with, and assess the value of, direct-recording spectrographs in the determination of trace elements. N. E.

1756. **Isolation and identification of non-metallic inclusions with the method of direct chlorination.** M. Mandl, M. Kaše, R. Freiwillig and J. Dostál (Res. Inst. Iron Metall., Prague). *Hutn. Listy*, 1959, **14** (7), 617–620.—The components of the sample of oxide inclusions, isolated by chlorination, are identified as follows—SiO_2 by evaporation with HF in the presence of oxalic acid, Fe, Ti, V and Zr by separation with cupferron (extraction with $CHCl_3$), FeO colorimetrically with thioglycollic acid, TiO_2 colorimetrically with sulphosalicylic acid, vanadium oxides colorimetrically with diphenylamine, ZrO_2 gravimetrically as pyrophosphate, Al_2O_3 colorimetrically with 8-hydroxyquinoline, MnO colorimetrically after oxidation with KIO_4 in a medium of $HClO_4$, H_3PO_4 and HNO_3, Cr_2O_3 colorimetrically with diphenylcarbazide, and CaO complexometrically with fluorexone (calcein) as indicator. Detailed procedures are given. J. ZÝKA

1757. **Photometric determination of low contents of boron in alloyed steel.** L. Brháček and K. Kunzová (Res. Inst. VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1959, **14** (8), 710–714.—A new modification of the 1:2:5:8-tetrahydroxyanthraquinone method is described. *Preparation of the calibration curve*—To 0·4 g of boron-free steel add 0, 10, 20, or 30 μg of B (for ⩾0·0075% of B; for ⩾0·00375% of B only one-half of these amounts), H_2O (1·5 ml), and conc. H_2SO_4 (0·8 ml) and dissolve on a water bath in a quartz flask fitted with a quartz reflux-condenser. Add H_2O_2 (10%) (3 ml) in small portions and heat for 10 min. Filter, dry and ignite the filter-paper with the undissolved residue and fuse with Na_2CO_3 (0·5 g) in a platinum dish. Dissolve the residue in few drops of H_2SO_4 (1:1) and add the soln. to the filtrate. Transfer to an electrolysis cell having a mercury cathode, dilute to 150 ml with H_2O and electrolyse at 0·25 amp. per sq. cm for 30 min. Filter into a platinum dish, evaporate to 5 ml (infra-red lamp), evaporate again to 3 ml in a smaller platinum dish, transfer to a 50-ml flask, and add sulphite soln. (10%) (0·3 ml). For ⩾0·0075% of B, dilute to vol., filter into a beaker, measure 2 ml into another flask, add 1:2:5:8-tetrahydroxyanthraquinone (**I**) soln. (25 ml) (0·120 g of **I** in 1 litre of conc. H_2SO_4; dilute 25 ml of this soln. with conc. H_2SO_4 to 500 ml). For ⩾0·00375% of B add conc. H_2SO_4 (5·5 ml), dilute to vol., centrifuge, measure 5 ml into a beaker and add **I** soln. (25 ml). Then cool, and after 60 min. measure the extinction with filter No. 607. The same procedure is used for the samples. Procedures for the determination of soluble and insoluble boron are given.

J. Zýka

1758. **Determination of carbon and sulphur in steel and cast iron by high-frequency combustion.** B. Bieber, B. Vejmělek and Z. Večeřa (Res. Inst. Materials and Technol., Brno, Czechoslovakia). *Hutn. Listy*, 1959, **14** (8), 700–706.—The procedure is carried out in a Siemens-Austria apparatus. The finely divided sample is burnt in a high-frequency field in a quartz combustion tube. Rose's metal or tin is added to the sample. Carbon is then determined gasometrically and sulphur iodimetrically.

J. Zýka

1759. **Determination of phosphorus in vanadium steel.** Katsumi Aiba (Shin-hokoku Steel Co., Kawagoe, Saitama-Ken). *Japan Analyst*, 1959, **8** (6), 394–395.—In the presence of V, the determination of P by the JIS (Japan Ind. Standard) method [pptn. of ammonium molybdophosphate in the presence of $(NH_4)_2SO_4.FeSO_4$] gives low values, even when an apparently pure yellow ppt. is obtained.

K. Saito

1760. **Rapid photometric determination of niobium in iron and steel by the molybdenum blue method.** Kimio Mukaewaki (Nippon Tokushu Steel Tube Co., Funato, Itabashi-ku, Tokyo). *Japan Analyst*, 1959, **8** (4), 219–225.—Davydov's molybdate method (*Zavod. Lab.*, 1947, **13**, 1038) was examined and a procedure for the routine determination of 0·05 to 1% of Nb is proposed. The decomposition of molybdophosphate and molybdoarsenate in 1·5 to 3 *N* H_2SO_4 is complete within 20 sec., whilst that of molybdoniobiophosphate begins 35 sec. after the addition of the acid. There is no interference from Ta (<5 mg for 0·5 mg of Nb), V (<0·05 mg), Cr (<30 mg), Ti (<1 mg), W (<50 mg), As (<2 mg) or Fe (<0·5 g). *Procedure*—Dissolve the sample (0·5 to 1 g) in HNO_3 (5 ml) and HF (45%) (5 ml), heat to white fumes with H_2SO_4 (1:2) (10 ml), cool, dissolve in water (10 ml) and, after the addition of HF (1:5) (5 ml), make up to 100 ml with water. Mix a 10-ml portion with water (10 ml), Na_2HPO_4 soln. (0·06%) (2·5 ml) and $(NH_4)_6Mo_7O_{24}$ soln. (2%) (5 ml), and maintain at room temp. for 15 min. Then add H_2SO_4 (1:2) (10 ml) and, after exactly 30 sec., $SnCl_2$ soln. (0·5% in 0·3 *N* HCl) (4 ml). Dilute to 50 ml with water and measure the extinction at 700 mμ.

K. Saito

1761. **Determination of chromium and vanadium in tungsten steel.** S. Arribas Jimeno (Dept. of Anal. Chem., Univ. of Oviedo, Spain). *Quim. e. Ind.*, 1959, **6** (4), 131–134.—A rapid method has been developed for the simultaneous determination of V and Cr in tungsten steel, without separation of the W, which is retained in solution as a complex with H_3PO_4. The steel is treated with H_2SO_4 - H_3PO_4; Cr^{3+}, V^{4+} and Mn^{2+} are oxidised with $(NH_4)_2S_2O_8$ in the presence of $AgNO_3$ as catalyst, the MnO_4^- are destroyed with NaN_3 and the sum of Cr and V is determined volumetrically with $(NH_4)_2SO_4.FeSO_4$ soln., with Ba diphenylamine-sulphonate (**I**) as indicator. The indicator is then destroyed, and the V^{4+} are selectively re-oxidised with $KMnO_4$, and the V^{5+} are determined volumetrically with $FeSO_4$ soln., with **I** as indicator. A method for determining V alone, in which the W is complexed with HF, is also described.

L. A. O'Neill

1762. **Solvent extraction by the use of radio-isotopes. II. Extraction of cobalt with thiocyanate.** Nobuo Suzuki and Shunichiro Muroi (Chem. Dept., Fac. Sci., Tohoku Univ., Katahira-cho, Sendai). *Japan Analyst*, 1959, **8** (5), 287–291.—When a mixture of *iso*amyl alcohol and ethyl acetate (1:5) is used, the extraction of Co in the presence of NH_4SCN is not affected by pH (1 to 7). The rate of extraction increases with increase in concn. of SCN^-, acetone and indifferent salts in the aq. phase.

III. The extraction of the cobalt nitroso-naphthol complex. Nobuo Suzuki and Hiroyuki Yoshida. *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (9), 1005–1008.—The extraction was examined with ^{60}Co as tracer. In the presence of a large excess (>50 times molar) of 1-nitroso-2-naphthol (**I**) (0·1% in CCl_4) (5 ml), the yield is >95% at pH 3·5 to 8·5. The optimum pH range decreases with decrease in the excess of **I**. The extraction yield is only slightly affected by ionic strength in the acid region, but markedly increases with increase in ionic strength in the basic region. The use of 2-nitroso-1-naphthol (0·1% in CCl_4) (5 ml) slightly increases the optimum pH range (2·5 to 8·5).

IV. Extraction of cobalt 1-nitroso-2-naphthol with carbon tetrachloride. Nobuo Suzuki and Hiroyuki Yoshida. *Ibid.*, 1959, **80** (9), 1008–1011.—The extraction is not affected by NH_4Cl and KH_2PO_4, but is retarded by EDTA. The effect of KCN depends on the pH, but the extent to which the extraction is retarded depends on experimental conditions, including shaking time and temp. Once extracted, however, the Co - **I** complex is not decomposed with EDTA or KCN. When the extraction yield is plotted against the molar ratio of **I** to Co, a break in the curve occurs at a ratio of 3.

K. Saito

1763. **Colorimetric determination of cobalt by means of its reaction with glycerol.** G. D. Nessonova, E. K. Pogosyants and M. O. Lishevskaya (Moscow Textile Inst.). *Zavod. Lab.*, 1959, **25** (7), 786–789.—By means of its reaction with glycerol in alkaline soln. Co can be determined in the presence of large amounts of Ni and Fe. To determine Co (20 to 88%) in alloys containing Ni, the sample (0·1 to 0·2 g) is dissolved in dil. HCl with the addition of HNO_3, the soln. is evaporated, the residue is dissolved in water and diluted to 100 ml, and an aliquot (10 ml) is mixed first with 20 ml of glycerol (sp. gr. 1·46) and then with 10 ml of 10 *N* NaOH. After 5 to 10 min. the extinction of the blue soln. is measured with a red filter.

G. S. Smith

1764. **Rapid colorimetric method of determining nickel in cobalt - arsenic ores.** Yu. N. Lushchik (Lab. of the Ores Department "Tuvakobal't"). *Zavod. Lab.*, 1959, **25** (7), 801–802.—The sample (0·5 to 2 g) is dissolved in HCl and HNO_3, the soln. is evaporated to dryness several times with HCl, and an aliquot (10 ml) of the filtered and diluted aq. extract (100 ml) is treated with 7 ml of 20% Na K tartrate in 5% NaOH soln., 5 ml of 3% $(NH_4)_2S_2O_8$ soln., and 5 ml of a 1% soln. of dimethylglyoxime in 5% NaOH soln., followed by 10 ml of 5% EDTA (disodium salt) soln. After a few minutes the soln. is diluted to 100 ml and Ni is determined colorimetrically.

G. S. Smith

1765. **Nicotinamidoxime as an analytical reagent. II. Spectrophotometric determination of cobalt with nicotinamidoxime.** K. K. Tripathi and D. Banerjea (Indian Ass. for the Cultivation of Science, Jadavpur, Calcutta). *Z. anal. Chem.*, 1959, **168** (6), 407–410 (in English).—Nicotinamidoxime gives a deep blue colour with Co in alkaline aq. ethanolic medium. The effects of ethanol concn., pH, reagent concn., rate of reaction and temperature on colour development are shown and the composition of the coloured complex is discussed. *Procedure*—An acid soln. of Co^{II}, freed from all other cations and a large excess of alkali-metal salts, is treated with an excess of aq. reagent soln. Ethanol (40 ml per 100 ml of the final volume) and water are added and the pH is adjusted to about 10·8, the final volume then being made up with water. The extinction of the soln. is measured at 580 mμ against a water blank. The sensitivity of the method is 0·01 μg of Co per sq. cm, and the visual identification limit is 0·5 μg of Co per ml. The optimum range of concn. is 2 to 16 p.p.m. Within the optimum pH range (10·5 to 11·0) the molar ratio of reagent to Co should be <300. Most anions and cations interfere, but chloride, nitrate, acetate, citrate and tartrate are without effect. Cyanide and EDTA bleach the colour.

S. M. Marsh

1766. **Determination of nickel in crude copper by alternating current polarography.** Kamihiko Itsuki, Fukunosuke Suzuki and Fumio Sato (Osaka Refinery, Mitsubishi Metal and Mining Co., Shinkawasaki-cho, Kita-ku, Osaka). *Japan Analyst*, 1959, **8** (4), 239–245.—Nickel (<0·1%) is determined by automatic balancing a.c. polarography in a supporting electrolyte containing *M* KCN, 1·5 *M* NaOH and 4% of Na_2SO_3, in the presence of a large amount of Cu, under the same instrumental conditions as for Pb (Itsuki, *Ibid.*, 1958, **7**, 253). Lead affects the wave height of Ni, but to a constant extent in the presence of >20 mg of Pb per 50 ml. There is no interference from Ag, As, Sb, Fe, Se or Zn. A large excess of HNO_3 vitiates the determination. *Procedure*—Dissolve the sample (5 g) in HNO_3 (1:1) (40 ml), dilute to 100 ml and to a 10-ml portion add $Pb(NO_3)_2$ (=20 mg of Pb), NaOH soln. (7·5 *M*) (10 ml), KCN soln. (5 *M*) (10 ml) and Na_2SO_3 soln. (70%) (10 ml), and dilute to 50 ml. Measure the wave height of the a.c. polarogram (−0·5 to 0·9 V, superimposed potential 20 mV).

K. Saito

1767. **Tiron as a spectrophotometric reagent for the estimation of osmium.** A. K. Majumdar and C. P. Savariar (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **21** (2), 146–150.—Tiron reacts with Os^{IV} in acetate-buffered soln. (pH 4·9 to 5·5) to give a red - violet colour (max. at 470 mμ). To the sample soln. (50 to 750 μg of Os) are added 5 ml of tiron soln. (5%), 5 ml of Na acetate soln. (5 *N*) and water to 20 ml. After heating for 15 min. on a steam bath the soln. is cooled, diluted to 25 ml and the extinction is measured in a 1-cm cell at 470 mμ (1·0 for 35 p.p.m. of Os). Ferric Fe, Ti^{4+}, Nb^{5+}, Ta^{5+}, Ce^{4+}, UO_2^{2+}, CrO_4^{2-}, MoO_4^{2-}, VO_3^- and the platinum metals other than Pt interfere. Silver and Au are reduced to metal, Pt can be tolerated up to 10 times the Os content and Al^{3+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Sn^{4+}, Pb^{2+}, Zr^{4+}, Th^{4+}, Hg^{2+} and WO_4^{2-} are "tolerated to a limited extent." Beer's law is obeyed in the range from 2 to 32 p.p.m. of Os.

T. R. Andrew

1768. **Rapid method for determination of the basicity of Siemens - Martin slags.** H. vom Ende and F. Bardenheuer (Mannesmann A.-G., Hüttenwerk Huckingen). *Arch. Eisenhüttenw.*, 1959, **30** (7), 391–396.—The determination is carried out by measurement of the electrical conductivity of aq. suspensions of powdered slag. The effects of period of stirring, temp., particle size and concn. were investigated, and conditions are specified that enable the basicity to be determined within 6 min. of sampling. The conductivities of aq. suspensions of blast-furnace and Thomas slags were also studied.

H. Sawistowski

1769. **Quantitative determination of the four major phases of Portland cement by combined X-ray and chemical analysis.** L. E. Copeland, S. Brunauer, D. L. Kantro, E. G. Schulz and C. H. Weise (Portland Cement Ass. Res. and Devel. Lab., Stokie, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1521–1530.—A method is described for determining the amounts of tricalcium silicate, β-dicalcium silicate, tricalcium aluminate and ferrite. The results are compared with those obtained by the Bogue method and by microscopical analysis.

K. A. Proctor

See also Abstracts—**1638**, Spot reagents for metals. **1639**, Trace metals in dyestuff intermediates. **1642**, Analysis by phosphor poisoning. **1644**, Maleanilic acid as reagent. **1650**, Free acids in presence of hydrolysable cations. **1817**, Analysis of fuel gases. **1863**, Detection of iodide. **1931**, Titanium in wine. **1980**, Automatic H_2O indicator for clay. **1986**, Determination of NH_4. **2018**, Lead in ores. **2028**, Analysis of metal films by neutron activation.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

1770. **Empty-tube combustion methods for the elementary micro-analysis of organic compounds.** J. E. Fildes (Dept. of Med. Chem., Australian Nat. Univ., Canberra). *Rev. Pure & Appl. Chem.*, 1959, **9** (2), 117–137.—Empty-tube combustion methods described and recommended by other workers for the determination of C, H, S, Cl, Br, I, F, P and Si are critically reviewed. Methods for the determination of several elements simultaneously are discussed. (64 references.) E. J. H. BIRCH

1771. **Quantitative organic elementary micro-analysis.** H. Wagner (Micro-anal. Lab., J. R. Geigy A.-G., Basle). *Chimia*, 1959, **13** (7), 213–219.—A review of the recent literature is presented. The determination of halogens, S, O, N, C and H is discussed in detail. (62 references.) H. M.

1772. **Nitrogen combustion train ((micro-Dumas). Microchemical apparatus.** British Standards Institution (2 Park Street, London, W.1). B.S. 1428 : Part A2: 1959. 15 pp.—Components are specified and illustrated (7 fig.).

1773. **Gas-chromatographic investigation of the nitrometer gas evolved during the determination of nitrogen by the Dumas method.** G. Kainz and F. Kasler (II Chem. Inst., Univ. Wien, Austria). *Z. anal. Chem.*, 1959, **168** (6), 425–429 (in German).—Gas chromatography has indicated the presence of methane as an impurity in nitrometer gases obtained during the Dumas determination of N. The amount of methane present depends on the oven temperature and the rate of gasification. Sterols and aliphatic hydrocarbon derivatives are particularly liable to give methane. At 700° only the Pregl method of individual combustion gives methane-free nitrometer gas. Automatic combustion at this temperature results in considerable contamination with methane, but at higher temperatures, 850° to 900°, the gas becomes methane-free. S. M. MARSH

1774. **Rapid determination of organically bound fluorine.** B. Z. Senkowski, E. G. Wollish and E. G. E. Shafer (Hoffman-La Roche Inc., Nutley, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1574–1576.—The sample is burned with Na_2O_2 in an atmosphere of oxygen, in a Schöniger borosilicate-glass flask. The combustion products are dissolved in water and the fluorine is determined spectrophotometrically with ZrO^{2+} - Eriochrome cyanine R (C.I. Mordant Blue 3) soln. (*cf.* Megregian, *Anal. Abstr.*, 1954, **1**, 2676). The standard deviation is about $\pm 0{\cdot}1$ from 37 determinations and the over-all accuracy is within $\pm 2\%$. Large quantities of PO_4^{3-} interfere, but can be removed by a Willard - Winter distillation. G. P. COOK

1775. **Determination of chlorine in organic liquids.** A. K. Santra and N. G. Banerjee (Central Fuel Res. Inst., Jealgora, Bihar). *J. Instn Chem., India*, 1959, **31** (3), 103–108.—The apparatus and procedure are described for the determination of chlorine in highly volatile organic liquids. The vapour of the sample is passed in a stream of N through a column of pptd. MnO_2 at 450°. The exit gases are bubbled through NaOH soln. containing some $NaHSO_3$, and after the oxidation the MnO_4^- is washed into this absorbent soln. Unchanged MnO_4^- is then reduced with oxalic acid, the soln. is filtered and the chlorine is determined in an aliquot, either volumetrically or gravimetrically. C. A. SLATER

1776. **Quantitative micro-determination of functional groups in organic compounds.** W. Schöniger (Micro-anal. Lab., Pharm. Chem. Div., Sandoz A.-G., Basle). *Chimia*, 1959, **13** (7), 220–224.—The determination of alkoxyl, acyl, C-methyl and carbonyl groups is discussed, and recent modifications to established methods together with newer methods are described. (30 references.) H. M.

1777. **Micro-determination of keto groups by the method of oxime formation.** V. A. Klimova and K. S. Zabrodina. *Izv. Akad. Nauk SSSR*, 1959, (7), 1343–1345.—*Procedure*—Dissolve 0·7 g of hydroxyammonium chloride (**I**) in H_2O (10 ml), add triethanolamine (0·5 g) and dilute the soln. to 100 ml with 96% ethanol; heat the flask (fitted with an air-condenser) containing this soln. (5 ml) and the test substance (3 to 10 mg) at 70° to 75° for 1 to 3 hr., cool, add 0·1% bromophenol blue soln. (3 drops) in 20% ethanol and back-titrate the excess of triethanolamine with 0·2 *N* HCl. Potentiometric titration is recommended when the soln. is coloured or cloudy. With 0·1 *N* **I** the accuracy of the method is within $\simeq 0{\cdot}3\%$ and with 0·5 *N* **I** the accuracy is reduced to $\simeq 0{\cdot}5\%$. K. R. C.

1778. **Organic micro-analysis. V. Improved scrubbing reagent for the determination of alkoxyl groups.** Minoru Fukuda (Pharm. Fac., Osaka Univ., Hotarugaike, Toyonaka). *Japan Analyst*, 1959, **8** (6), 387–390.—The preparation of a modified, more active, soda lime reagent for use in the Pregl method (*cf.* Friedrich, *Mikrochem.*, 1929, **7**, 185) is described. Commercial soda lime, KOH and water (20:60:3, w/w) and NiO (a very small amount) are fused, crushed and sieved, the fraction between 8 and 25 mesh being collected.

VI. Improvement in the micro-determination of alkoxyl groups. Minoru Fukuda. *Ibid.*, 1959, **8** (6), 390–393.—Kirsten's apparatus (*Mikrochim. Acta*, 1955, 786) was modified so that the decomposition can be carried out in a sealed vessel, which is also used as the distilling flask. The outlet gas is scrubbed with the modified soda lime and determined by the combustion method (Fukuda and Sai, *Anal. Abstr.*, 1959, **6**, 3555). K. SAITO

1779. **Analytical applications of vapour-phase chromatography. II. Analysis of hydrocarbon mixtures.** G. Caprioli, E. Pavan and M. De Vita (Lab. Sicedison, Mantova, Italy). *Ann. Chim., Roma*, 1959, **49** (7–8), 1120–1124.—The development of columns, with special reference to mixtures of polar and non-polar stationary phases, for the separation of C_1 to C_4 hydrocarbons, including olefinic and acetylenic derivatives, has been studied. Good separations are achieved with a mixture of butyrolactone and liquid paraffin on Celite and alumina. The correction factors necessary for quantitative determinations have been measured. L. A. O'NEILL

1780. **Gas-chromatographic separation of 3- and 4-methylpent-1-ene.** B. Smith and R. Ohlson

(Inst. f. Org. Kem., Chalmers Tek. Högsk., Göteborg, Sweden). *Acta Chem. Scand.*, 1959, **13** (6), 1253.—The separation of 3- and 4-methylpent-1-ene can be achieved by gas chromatography by using as stationary phase one part of ethanediol satd. with $AgNO_3$ adsorbed on two parts of 30 to 60-mesh Chromosorb, and packed in two aluminium tubes each 2 metres × 4 mm. The carrier gas used is He at 50 ml per min. at 25°. E. J. H. BIRCH

1781. **Determination of dichlorodifluoromethane in fumigation mixtures with ethylene oxide by measurement of thermal conductivity.** W. A. Affens, E. O. Haenni and R. A. Fulton (U.S. Dept. Agric., Beltsville, Md., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1565–1568.—Ethylene oxide and water vapour are the chief interfering substances in the determination of dichlorodifluoromethane (**I**) by thermal conductivity when dry air is used as reference. After their removal the thermal-conductivity method can be readily applied with commercially available instruments. Moisture is removed from the gas sample with anhydrous $CaSO_4$ and the ethylene oxide by the Lubatti technique (*J. Soc. Chem. Ind.*, 1935, **54**, 4248), in which the gas sample is passed through traps containing *N* H_2SO_4 saturated with MgBr. The ethylene oxide reacts with the acid, the excess of which is back-titrated with standard alkali soln. The accuracy for the ethylene oxide determination is generally within ±2% and that for **I** is within ≃±8%.
G. P. COOK

1782. **Colorimetric determination of organic peroxides.** M. I. Eiss and P. Giesecke (American Cyanamid Co., Stamford, Conn., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1558–1560.—Benzoyl-leucomethylene blue reacts with peroxides and hydroperoxides in benzene - trichloroacetic acid soln. to form the characteristic methylene blue colour. The reaction is sensitive to u.v. light and, to a less extent, to artificial light and heat. Of the five compounds tested, four (benzoyl peroxide, lauroyl peroxide, *p*-menthane hydroperoxide and cumene hydroperoxide) followed Beer's law at concn. up to 1 p.p.m. and one (*tert.*-butyl hydroperoxide) deviated slightly. The precision (95% confidence limits) ranged from ±1·7 to ±2·6%, and as little as 0·5 mg of active oxygen could be detected.
G. P. COOK

1783. **New method for the oxidation of methanol and for its colorimetric and polarographic determination.** J. Kubis (Chem. Inst., Komenský Univ., Bratislava, Czechoslovakia). *Čas. Lék. Čes.*, 1959, **98** (27), 851–856.—Methanol or ethanol can be oxidised to the corresponding aldehyde in a simple apparatus by means of granulated $PbCrO_4$ at 600°. For following the course of the oxidation quantitatively, the colorimetric method with Schiff reagent was used, which enables 0·5 to 3 mg of methanol per 100 ml to be determined. The polarographic determination of the formaldehyde formed by the oxidation of methanol can also be carried out with 0·1 *N* LiOH as electrolyte. J. ZÝKA

1784. **Determination of vinyl ethers and other unsaturated compounds. Modified mercuric acetate procedure.** J. B. Johnson and J. P. Fletcher (Union Carbide Chemicals Co., South Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1563–1564.—The sample is treated with an excess of mercuric acetate in methanol soln., to form the Hg addition compound and acetic acid. With vinyl ethers the reaction is conducted at −10°. Solid NaBr is added to convert the excess of Hg acetate into the bromide and the acetic acid is then directly titrated with alcoholic KOH soln. Data for the determination of 32 compounds are given. The standard deviation is ±0·2%. α,β-Unsaturated acids, aldehydes, esters, ketones and nitriles do not react quantitatively under the conditions employed, and inorganic salts, especially halides, must be absent.
G. P. COOK

1785. **Infra-red analysis of a mixture of 1:4-dihydroxy C_4's.** H. L. Spell (Dow Chemical Co., Freeport, Tex.). *Anal. Chem.*, 1959, **31** (9), 1600.—Data for but-2-yne-1:4-diol, *cis*-but-2-ene-1:4-diol and butane-1:4-diol are given.

1786. **The analysis of 1:2-glycols and polyhydric compounds. I. Indirect iodimetric determination of ethanediol, glycerol or mannitol by means of the formaldehyde formed by oxidation with periodate.** L. Maros and E. Schulek (L. Eötvös Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (3), 359–364 (in German); *Magyar Kém. Foly.*, 1959, **65** (9), 361–363 (in Hungarian).—The test soln., 10 ml containing 0·75 to 0·85 g of ethanediol (**I**), 1·2 to 1·4 g of glycerol (**II**) or 2·3 to 2·5 g of mannitol (**III**) per 500 ml, is treated with 2, 3 or 5 ml (for **I**, **II** and **III**, respectively) of 0·3 *M* HIO_4. Then 1·5 *M* Na_2SO_3 is added until the brown or yellow colour is discharged, followed by a further 0·5 ml. The mixture is neutralised to methyl red with 10% acetic acid and acidified by addition of a further 1 ml of acid. Pentane (5 ml) is introduced to prevent atmospheric oxidation and after 10 min. the excess of sulphite is removed by titration with 5% or 0·1 *N* iodine, with starch soln. as indicator. In order to decompose the formaldehyde bisulphite, the mixture is made alkaline by the addition of 3 ml of 20% NaOH soln., and treated with 0·2 to 0·3 g of KCN, with shaking. After 3 to 4 min. the soln. is neutralised and then acidified with 1 ml of HCl and titrated with 0·1 *N* iodine, with starch soln. as indicator, until a permanent blue colour is obtained. The amount of formaldehyde, and hence the amount of hydroxy compound, is calculated. Typical results are tabulated. S. M. MARSH

1787. **Paper ionophoresis of carbohydrates in molybdate solutions.** E. J. Bourne, D. H. Hutson and H. Weigel (Chem. Dept., Royal Holloway Coll., Englefield Green, Surrey, England). *Chem. & Ind.*, 1959, (33), 1047–1048.—Ionophoresis of a number of carbohydrates is carried out on Whatman 3MM paper at 15 V per cm in 0·1 *M* sodium molybdate adjusted to pH 5 with H_2SO_4. The positions of the compounds were revealed with acetone silver nitrate - alcoholic NaOH (Trevelyan *et al.*, *Nature*, 1950, **166**, 444). Results are expressed as a comparison with the migration of D-glucitol, with glycerol as a non-migrating marker. The separation of some pentoses and hexoses from their corresponding alcohols is effected. Of the aldohexoses and aldopentoses examined, only those with a *cis-cis*-1:2:3-triol system (which can adopt the conformation of two axial and one equatorial hydroxyl group) migrate. Because of the difference in the number of Mo atoms complexed, oligosaccharides fall into three groups, *viz.* those with 1:3 glycoside linkages (no migration), those with 1:4 linkages (medium migration), and those with 1:2 or 1:6 linkages (migration ≃0·8 to 0·9 compared with D-glucitol). E. J. H. BIRCH

1788. **Reaction of sodium chlorite with various polysaccharides. Rate studies and aldehyde-group determinations.** H. F. Launer and Yoshio Tomimatsu (West Reg. Res. Lab., Albany, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1569–1574.—The aldehyde contents and degree of polymerisation of some polysaccharides were calculated from the chlorite results, obtained under various conditions of pH and temp., and using stoicheiometric ratios previously determined for substances of low mol. wt. The results with the various chlorite procedures (*Ibid.*, 1954, **26**, 382; 1959, **31**, 1385; *J. Amer. Chem. Soc.*, 1954, **76**, 2591) were self-consistent, and agreed well with those obtained by other methods. G. P. COOK

1789. **Chromatography of organic compounds. II. Paper-chromatographic separation of ketones and aldehydes as rhodanine derivatives.** J. Franc (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1959, **24** (7), 2102–2108.—The condensation reaction of aldehydes and ketones with rhodanine was used for their chromatographic separation. The relation between the structure and the chromatographic behaviour of the compounds studied was investigated. *Procedure*—Dissolve the sample of aldehyde or ketone (0·1 g) and rhodanine (0·1 g) in aq. NH_3 (0·5 ml) and add ethanol till the soln. is clear. Add NH_4Cl soln. (10 g in 20 ml of hot H_2O) (1 ml) and heat the mixture under reflux for 15 min. Separate the products chromatographically on Whatman No. 1 paper impregnated with a 30% ethanolic soln. of formamide or dimethylformamide, with chloro-*cyclo*hexane, CCl_4, chlorobenzene, or a mixture of *cyclo*hexane with butanol (25:1) as mobile phase. For the detection use u.v. light or a 5% soln. of $AgNO_3$ in aq. NH_3. J. ZÝKA

1790. **New colour reaction for the detection of organic acids on paper chromatograms.** K. Schlögl (II Chem. Inst., Univ. of Vienna, Austria). *Naturwissenschaften*, 1959, **46** (14), 447.—The disodium or diammonium disulphonate of dihydroindanthrene is sprayed on paper chromatograms and converted by any acid present into indanthrene. The excess of reagent is easily washed out with dil. NaOH soln. and water. The residual blue spots are readily discerned and very stable. The disodium deriv. is used in 0·01 to 0·02% aq. soln., but the diammonium deriv. may be used in solvent mixtures [*e.g.*, *n*-butanol - ethanol - conc. aq. NH_3 - H_2O (4:4:4:1 or 4:4:1:3)], though the paper must be dried for 20 to 30 min. at 120° to 140° before spraying. The detectable limit of benzoic acid is 20 μg and the optimum amount is 50 to 100 μg. The method appears to be universally applicable for the detection of acids; R_F values are given for 12 acids. J. P. STERN

1791. **Determination of volatile fatty acids with paper chromatography.** D. Hal'ama (Dept. of Tech. Microbiol. and Biochem., Inst. Chem. Technol., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1959, **13** (7–8), 454–463.—After distillation the fatty acids are converted into hydroxamic acid derivatives, then chromatographed, and the spots are detected with $FeCl_3$ soln. and determined photometrically. A new mathematical method for the calculations from the measurements is given. *Procedure*—To the sample (3 to 5 ml) in the distillation flask add stearic acid (to prevent bumping), H_2SO_4 (1:1) (0·5 ml), $MgSO_4.7H_2O$ (3 g) and formaldehyde (40%) (0·2 ml) (to prevent the distillation of SO_2). Distil off the volatile acids and titrate the distillate (100 to 130 ml) with 0·05 *N* NaOH (phenolphthalein as indicator). Add *N* NaOH (1 ml) and evaporate to dryness at 110°. To the residue add a satd. soln. of $KHSO_4$ (2 ml) containing a 25% excess of $KHSO_4$, and diethyl ether (10 ml). Extract by stirring for 1 min., set aside and transfer the supernatant liquid (5 ml) into a tube containing methanol - conc. H_2SO_4 (100:1) (1 ml). Close with a rubber stopper and set aside for 24 hr. Then add a mixture of 1·67 *N* hydroxyammonium chloride and 7 *N* Na methoxide (1:1) (2 ml) (mixed at 0°) and after 1 hr. chromatograph 5 to 10 μl of the liquid on Whatman No. 1 or 4 paper with *n*-propanol - acetic acid - H_2O (8:1:1) as solvent. Detect with a 5% soln. of $FeCl_3$ in methanol - acetone (4:3) and measure the separated spots by means of a photometer and green filter. J. ZÝKA

1792. **Sensitive method for the determination of sorbic acid.** H. Schmidt (Bundesforschungsanst. f. Lebensmittelfrischhaltung, Karlsruhe). *Naturwissenschaften*, 1959, **46** (13), 425.—Heat the sample with acid dichromate soln., allow the products to react with thiobarbituric acid in aq. soln. and measure the extinction at 532 mμ. Beer's law is obeyed in the range 1 to 20 μg of sorbic acid per ml. A. R. ROGERS

1793. **Manganimetry. IV. Oxidimetric determination of oxalic, malonic, tartaric, citric and salicylic acids with manganic pyrophosphate.** Masayoshi Ishibashi, Tsunenobu Shigematsu and Shozo Shibata (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1959, **8** (6), 380–382.—When heated with a large excess of standard manganic pyrophosphate soln. in 1·2 *N* H_2SO_4 for 15 min. on a water bath, oxalic acid is oxidised to CO_2 and H_2O, whereas malonic, tartaric, citric and salicylic acids are only partially oxidised, giving reproducible empirical equivalents of 4·34, 6·64, 8·60 and 11·60, respectively. Formic and acetic acids are not oxidised under the given conditions. The excess of Mn^{III} is reduced with a known amount of Fe^{II}, the excess of which is titrated with standard Mn^{III} soln., with diphenylamine as indicator. K. SAITO

1794. **Double-bond determination in acrylic esters by complexometric titration.** U. Bartels and H. Hoyme (Dtsch. Akad. d. Wissensch., Berlin). *Faserforsch. u. Textiltech.*, 1959, **10** (7), 345–347.—A rapid and accurate method for determining the purity of acrylic esters, also applicable to allyl alcohol but not to vinyl cyanide, depends on the formation of a complex with a mercuric salt, removal of the excess of Hg with an excess of EDTA, and back-titration with $ZnSO_4$ soln. *Reagent* (I)—Dissolve 44 g of mercuric acetate in 400 ml of water, add 5 ml of glacial acetic acid and 400 ml of dioxan, and dilute to 1 litre with water. *Procedure*—Add the sample (70 to 100 mg) to 5 ml of **I**, set aside for 1 hr. with occasional shaking, and dilute to 100 ml with water. To an aliquot (20 ml) add 5 ml of buffer soln. (pH 10) (550 ml of conc. aq. NH_3 and 54 g of NH_4Cl made up to 1 litre with water) and 20 to 50 ml of 0·2 *M* EDTA, taking care that any initial ppt. is completely re-dissolved. Then add Eriochrome black T (C.I. Mordant Black 11) indicator, and titrate the green soln. to a red - brown end-point with 0·2 *M* $ZnSO_4$. The results agree well with those by the pyridine sulphate - dibromide method (Rowe *et al.*, *Anal. Chem.*, 1944, **16**, 571) and it is shown that the method of Marquardt and Luce (*Anal. Chem.*, 1948, **8**, 751) is unsatisfactory. H. L. WHITEHEAD

1795. **Paper chromatography in the analysis of polyesters. II.** I. Arendt and H.-J. Schenck (Siemens-Schuckertwerke, Kabelwerke, Berlin-Siemensstadt). *Kunststoffe*, 1959, **49** (7), 321–322.—A previously published method (*cf. Anal. Abstr.*, 1958, **5**, 4222) involves a preliminary saponification with ethanolic KOH. In the presence of fumaric acid this yields a small amount of ethoxysuccinic acid. To avoid interference from this, a new solvent system for dicarboxylic acids is advocated [benzene - acetone - formic acid - water (10:5:2:6)] in which fumaric and ethoxysuccinic acids have the same R_F value. R_F values for 8 dicarboxylic acids are given. C. A. FINCH

1796. **Application of chelatometry to the volumetric determination of diacetyl.** C. Hennart and E. Merlin (Orgafrance, Lab. de Villiers-Saint-Georges, Seine et Marne, France). *Chim. Anal.*, 1959, **41** (7), 287–289.—Diacetyl is converted into its dioxime, which is pptd. with standard nickel soln., and the excess of Ni is determined by titration with a soln. of EDTA (disodium salt), with murexide as indicator. *Procedure*—The sample containing a weight of diacetyl $\equiv \simeq 50\%$ of the nickel added is weighed into a flask containing 25 ml of *N* hydroxyammonium chloride and 25 ml of 0·2 *M* Ni acetate and allowed to boil gently under reflux for 4 hr. The flask is cooled and, after the addition of 50 ml of aq. NH_3 soln., the contents are made up to 250 ml. The excess of Ni is determined on a filtered 25-ml aliquot by titration with 0·02 *M* EDTA (disodium salt), with murexide as indicator. The method has been applied successfully to pure diacetyl and to soln. in ethanol and in ethyl methyl ketone. E. J. H. BIRCH

1797. **Product-analysis of the gas-phase nitration of methane. Determination of nitromethane in the presence of nitrite.** J. Giber and T. Meisel (Dept. Ind. Org. Chem., Tech. Univ., Budapest). *Magyar Kém. Foly.*, 1959, **65** (7), 260–263.—By this method the analysis can be carried out without separating the constituents of the mixture. Nitromethane (**I**) is oxidised to NO_2^- and the concn. of **I** is determined from the difference between the NO_2^- concn. before and after oxidation. *Procedure*—Dilute the sample (2 ml) with H_2O (20 ml), neutralise it with HCl, add dil. HCl (1:5) (1 ml) and 1% sulphanilic acid soln. (5 ml). Shake the flask for 1 min., then add a 1% soln. of hydroxynaphthoic acid (10 ml, containing 4% of NaOH) and dilute the soln. to 100 ml. After 10 min., determine the NO_2^- in a Pulfrich photometer with a green filter. Gently boil another aliquot (2 ml) under reflux with 50% NaOH soln. (2 ml) and 30% H_2O_2 (1 ml) for $\simeq 10$ min., until no more O is evolved. Dilute the cooled soln. to 100 ml and determine the NO_2^- in a neutralised aliquot as before. If a large excess of NO_2^- over **I** is present, add sufficient urea to destroy about 80% of the NO_2^- and keep the soln. at 30° for 20 min. To two aliquots (2 ml each) add 50% NaOH soln. (2 ml) and boil the soln. for $\simeq 10$ min. and determine the concn. of NO_2^- and **I** as before. This last method avoids the inaccuracy caused by calculating the concn. of **I** as a small difference between two large values. The relative error in both methods is 2 to 3%. Any mononitroparaffin can be determined similarly and the method can also be adapted for the determination of polynitroparaffins. The constituents of the nitrating mixture (including HNO_3) do not interfere. A. G. PETO

1798. **Conductimetric and potentiometric titrations of nitrogen bases in non-aqueous solvents.** N. van Meurs and E. A. M. F. Dahmen (Koninklijke/Shell-Lab., N.V. De Bataafsche Petroleum Maatschappij, Amsterdam, Netherlands). *Anal. Chim. Acta*, 1959, **21** (2), 193–202.—The titration of mono- and bi-valent bases by both potentiometric and conductimetric methods has been studied from the standpoint of the effect of the solvent, the titrant and temperature. The use of nitrobenzene as solvent and potentiometric titration with perchloric acid is advocated, especially for mixtures of bases. T. R. ANDREW

1799. **Determination of the hydrochlorides of tertiary amines in anhydrous medium, in the presence of the hydrochlorides of acetylatable amines.** I. Gyenes (Pharm.-technol. Lab. of the Pharm. Factory of Kőbánya, Budapest). *Magyar Kém. Foly.*, 1959, **65** (7), 264–276.—A detailed theoretical and practical description is given of the determination of a tertiary amine in the presence of acetylatable amines. The acetylating mixture converts the primary and secondary amines into amides which, under the experimental conditions, consume hardly any standard soln. *Procedure*—Dissolve the hydrochloride of the tertiary amine (0·3 to 0·5 milli-equiv.) in a mixture of acetic anhydride (40 to 80%) and acetic acid containing Hg^{II} acetate (0·3 g). Allow the acetylation to proceed at room temp. for 80 to 180 min. and then titrate the tertiary amine with 0·05 *N* $HClO_4$ in acetic acid. Depending on the basic strength of the amides formed, the indicator is crystal violet or cresol red. A. G. PETO

1800. **Application of zone electrophoresis and polarography to the analysis of complexone mixtures.** W. Hoyle and T. S. West (Univ., Birmingham, England). *Talanta*, 1959, **3** (1), 47–53.—A method for the separation of different polyaminocarboxylic acids (complexones) by paper electrophoresis of their copper chelates at pH 5 is described. The separated chelates may be eluted from the electropherogram and examined by polarography in 0·4 *M* acetic acid. In many cases the $E_{\frac{1}{2}}$ values and the electrophoretic migration rates serve to characterise the complexones. By use of a standard curve of observed $\Delta E_{\frac{1}{2}}$ values *vs.* known log *K* values, an estimate of the stability of the copper chelate may be obtained. G. P. COOK

1801. **Analysis of aromatic solvents by gas chromatography.** B. Smith (Inst. för Org. Kem., Chalmers Tek. Högsk., Göteborg, Sweden). *Acta Chem. Scand.*, 1959, **13** (5), 877–883 (in English).—A stationary phase of *p*-nitroaniline picrate (20% on Silocel C22) enables saturated hydrocarbons to be separated from commercial benzene by gas chromatography (110°; He at 20 ml per min. in two 1-metre columns). These saturated hydrocarbons are then condensed and further separated by using tetra-*n*-pentylsilane (3% on Chromosorb) (for the separation of *cyclo*hexane and 3:3-dimethylpentane) or squalane (3% on Chromosorb) (25°; He at 80 ml per min. in two 3-metre columns). For the separation of 2-methylhexane and 2:3-dimethylpentane a 6-metre di-*n*-octyl phthalate column is necessary. "Industrial pure toluol" is analysed on a *p*-nitroaniline picrate column. "Industrial pure xylol" is analysed with a 2-metre column of fluorene picrate (20% on Celite 545) (100°; He at 80 ml per min.), which does not separate benzene from the saturated hydrocarbons, or a 2-metre column of $\beta\beta'$-hydroxypropionitrile (20% on Chromosorb) (100°; He at 50 ml per min.), which does not effectively separate *m*- and

p-xylenes from ethylbenzene, or best with a *p*-nitroaniline picrate column. The separate determination of *m*- and *p*-xylenes is not possible by gas chromatography. Examples of analyses are given and it is considered sufficiently accurate to equate the percentage areas to the percentage by wt.

E. J. H. BIRCH

1802. **Analysis of benzene - mesitylene mixtures.** R. J. Jakobsen and C. D. Smith (Battelle Memorial Inst., Columbus, Ohio). *Anal. Chem.*, 1959, **31** (9), 1600.—Infra-red data for benzene and mesitylene are given.

1803. **Analysis of toluene - butylbenzene mixtures.** R. J. Jakobsen and C. D. Smith (Battelle Memorial Inst., Columbus, Ohio). *Anal. Chem.*, 1959, **31** (9), 1600.—Infra-red data for toluene and butylbenzene are given.

1804. **Chromatography of aromatic isomers. XI. Paper-chromatographic separation of hydrocarbons after nitration.** J. Franc and J. Knížek (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1959, **24** (7), 2299–2305.—Twenty-seven aromatic hydrocarbons were separated by paper chromatography, after nitration. The relation between constitution and the chromatographic behaviour was studied. Ascending paper chromatography was carried out on Whatman No. 1 paper, impregnated by washing in a 20% soln. of petroleum (boiling-range 190° to 275°) in light petroleum. Ethanol - acetic acid - H_2O (20:1:14) was used as mobile phase. The separated spots were detected with Ehrlich reagent after reduction with 10% $SnCl_2$ soln. R_F values of the compounds studied were determined.

J. ŽÝKA

1805. **Light-absorption studies. XIV. The ultra-violet absorption spectra of phenols.** J. C. Dearden and W. F. Forbes (Memorial Univ. of Newfoundland, St. John's). *Canad. J. Chem.*, 1959, **37** (8), 1294–1304.—The spectra of phenol and its *o*-, *m*- and *p*-substituted derivatives in various solvents are listed. The hydrogen bonding and steric effects are emphasised.

XV. The ultra-violet absorption spectra of anisoles. J. C. Dearden and W. F. Forbes. *Ibid.*, 1959, **37** (8), 1305–1314.—The spectra of anisole and its *o*-, *m*- and *p*-derivatives are similar to those of phenols except that properties determined mainly by the H atom of the OH group are modified.

N. E.

1806. **Gas chromatography applied to the analysis of phenols.** J. S. Fitzgerald (C.S.I.R.O., Melbourne). *Aust. J. Appl. Sci.*, 1959, **10** (2), 169–189.—Complex phenolic mixtures, *e.g.*, in tar acids, can be partly separated at ≃200° on spiral columns (8 to 12 ft. long), with either polar or non-polar stationary phases. The non-polar phase (preferably a spray-dried mixture of Na_2SO_4 and Na dodecylbenzenesulphonate) has a longer retention time for phenols of high b.p. (190° to 275°) when they have *o*-alkyl substituents, whilst the polar phase (preferably Apiezon-L on Celite) releases such phenols earlier than those of equal volatility but with no *ortho*-substitution. Complete resolution is obtained by using the two phases in series, peak eluates from the first (non-polar) column being fed to the second in successive runs (*cf.* Simmons and Snyder, *Anal. Chem.*, 1958, **30**, 32). Each fraction is thus resolved analytically on the second phase. The methylation of dihydric or chlorinated (but not alkyl) phenols may be advantageous. Retention times for ≃50 phenols (b.p. 190° to 275°) on different phases are listed. Dihydric phenols can be chromatographed with Silicone E–301 as stationary phase. Notes on 26 stationary phases are given.

W. J. BAKER

1807. **The reaction of phenol with formaldehyde. I. The separation and quantitative determination of low-molecular-weight reaction products by paper chromatography.** H. G. Peer (Central Lab., T.N.O., Delft). *Rec. Trav. Chim. Pays-Bas*, 1959, **78** (8), 631–643 (in English).—Phenol - formaldehyde reaction products containing up to 4 phenol nuclei per mol. can be separated by two-dimensional paper chromatography. The eluents used are benzene - acetic acid - water, which separates the components according to the number of *o*-hydroxyl groups per mol., and water containing CO_2, which separates them according to mol. wt. In this way *o*- and *p*-hydroxymethylphenols have been separated from each other and from the reaction mixture. The separated compounds can be extracted by elution with water if the strips containing the spots are covered with paper strips of the same size. The extract is diluted to 8 ml and 0·2% aq. 4-aminophenazone (0·6 ml), 0·4 *N* aq. NH_3 (1 ml) and 0·4% $K_3Fe(CN)_6$ soln. are added. The extinction of the red colour formed is measured at 510 mμ. Quantities of 0·005 to 0·1 mg of hydroxymethylphenols can be determined to within 2%.

G. BURGER

1808. **Quantitative determination of some polyhydric phenols.** G. R. Boreham and J. A. P. Cunningham (North Thames Gas Board, Fulham Research Lab., London). *Fuel, London*, 1959, **38** (4), 489–494.—A method has been developed for the determination of phenols in trace amounts in gas-works liquors in order to assess the efficiency of biological oxidation. The phenols, as aq. syrups or conc. ethanolic soln., are separated by paper chromatography with a mixture of benzene, acetic acid and H_2O (3:3:1, by vol.). Weighed and unweighed amounts are run in parallel. The developed qualitative chromatogram is sprayed with a 0·1% ethanolic soln. of 2:6-dichloro-*p*-benzoquinone-4-chlorimine and, after drying, with 10% Na_2CO_3 soln. The colours and R_F values obtained with 18 phenols are given. The corresponding areas of the quantitative chromatogram are cut out and the phenols extracted with ethanol. The absorption spectra of the extracts are recorded between 250 and 320 mμ, and the phenols are determined by reference to a calibration graph.

G. BURGER

1809. **Separation of acetopyrocatechol [3:4-dihydroxyacetophenone] from catechol and some 4-substituted catechol- or guaiacol-type phenols using chelation reactions.** J. Halmekoski (Inst. Chem., Univ. Helsinki, Finland). *Suomen Kem., B*, 1959, **32** (7–8), 170–172 (in English).—Paper-chromatographic and electrophoretic methods were devised for the separation of 3:4-dihydroxyacetophenone from other monomeric lignin-cleavage phenols obtained in the degradation of lignin (*e.g.*, vanillin, 4-hydroxy-3-methoxyacetophenone, 3:4-dihydroxybenzaldehyde, catechol and 3:4-dihydroxybenzoic acid). Owing to the formation of chelates, phenols of the catechol type move much more slowly than those of the guaiacol type on paper impregnated with Na_2MoO_4, sodium molybdophosphate, Na_2WO_4, $Na_2B_4O_7$, or $NaVO_3$. The former are generally separable by paper chromatography owing to differences in the stabilities of the

complexes and in the hydrophilic groups of the ligands. A complete separation of all the phenols studied is possible by the electrophoretic technique described. E. SJÖSTRÖM

1810. **Analysis of ionones and methylionones by gas - liquid partition chromatography.** R. O. Crisler and C. L. Benford (Procter & Gamble Co., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1516–1518.—The isomeric composition of commercial ionones and methylionones is determined by conventional gas - liquid chromatography with a column packed with Apiezon L on Celite. The accuracy is $\pm 1{\cdot}4\%$ and the reproducibility $\pm 0{\cdot}6\%$. Non-ionone impurities can also be detected. G. P. COOK

1811. **Colour reaction of polynitro compounds (Janovsky reaction).** T. Urbański, S. Kwiatowska and W. Kutkiewicz (Dept. Org. Technol., Tech. Univ., Warsaw). *Bull. Acad. Polon. Sci., Sér. Sci. Chim., Géol. et Géogr.*, 1959, **7** (6), 397–398 (in English).—Data on the absorption maxima and colours of the products of nine of the more common di- and tri-nitro derivatives of benzene and toluene are tabulated. A few crystals of the nitro compound are dissolved in acetone (50 ml); 30% aq. KOH soln. (2 to 3 ml) is added, the mixture is shaken and after 45 min. the acetone soln. is examined in a Unicam SP500 spectrophotometer. K. R. C.

1812. **Study of the sulphonation reaction. XLIX. Determination of isomeric toluenesulphonic acids in their mixtures.** A. A. Sprýskov. *Izv. Výssh. Uchebn. Zavedenil, Khim. i Khim. Tekhnol.*, 1958, (6), 42–46; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,239.—A method is developed for determining toluene-*m*-sulphonic acid (**I**) in a mixture of the isomers, based on the conversion of the sulphonic acids into sulphochlorides, nitration, saponification, reduction, and bromination, with a parallel determination of sulphuric acid and titration with a nitrite soln. To determine toluene-*p*-sulphonic acid (**II**), add to 5 or 6 g of the mixture, not containing toluene, 15 ml of chlorosulphonic acid at $<0°$, maintain for 1 hr. at 0°, and pour on to ice. Extract the mixture of sulphochlorides with $CHCl_3$, wash the extract, dry it with $CaCl_2$, remove the $CHCl_3$, and distil the residue *in vacuo* at $\simeq 150°$. Determine the content of **II** from the solidification curve of the sulphochlorides. The error is $<0{\cdot}8\%$. To determine **I**, to 1 g of the sulphochlorides add with cooling a mixture of 3 g of 100% H_2SO_4 and 2 g of HNO_3 (sp. gr. 1·52), stir for 30 min., allow the layers to separate and remove the mineral acids, extract the nitrosulphochlorides with $CHCl_3$, wash the extract with a soln. of Na_2CO_3, evaporate the $CHCl_3$, saponify the residue by heating with 10 ml of alcohol and distil off the alcohol. Dissolve the nitrotoluenesulphonic acids in water (5 ml), heat the soln. with active carbon and filter; to the filtrate add 20 ml of conc. HCl and, with cooling, 3 g of zinc dust; cool, filter, and make up to 100 ml. To 25 ml of the soln. add conc. HCl (5 ml), KBr (1 g) and 1 drop of a 0·2% soln. of Tropaeolin OO (C.I. Acid Orange 5) and titrate with 0·05 *N* $NaNO_2$; the total amount of toluenesulphonic acids is calculated from the formula—$A = 0{\cdot}00861\,a$, where a is the number of ml of 0·05 *N* $NaNO_2$. To another 25-ml aliquot of the same soln. add 5 ml of conc. HCl and excess of a 0·1 *N* soln. of KBr + $KBrO_3$, and set aside at room temp. for 30 min.; reduce the excess of Br with hydrazine hydrochloride ($\simeq 0{\cdot}05$ g), neutralise the soln. with aq. NH_3, add 1 ml of HCl, filter, and determine H_2SO_4 gravimetrically in the filtrate. The content of **I** in the mixture of isomers is calculated from the formula—$[(73{\cdot}77\,B)/A] - C$, where B is the wt. of $BaSO_4$ and C is the correction ($C = 0{\cdot}03/100 - 2B/A \times 73{\cdot}77$). The error in determining **I** is 1·4%. The content of toluene-*o*-sulphonic acid is found by difference. C. D. KOPKIN

1813. **Spectrophotometric determination of polycyclic hydrocarbons. II.** A. J. Lindsey (Dept. of Chem., Sir John Cass Coll., London, England). *Anal. Chim. Acta*, 1959, **21** (2), 101–104.—The absorption maxima of 23 polycyclic hydrocarbons are listed in the order of their elution from a chromatographic column (*cf. Anal. Chim. Acta*, 1959, **20**, 175). It is suggested that these wavelengths may be used to characterise the hydrocarbons and may permit of semi-quant. determination (*cf.* Karr, *Appl. Spectroscopy*, 1959, **13**, 15, 41). T. R. ANDREW

1814. **Organic analysis. XV. The infra-red spectra of the tetralin series.** Tsutomu Momose, Yo Ueda and Hiroshige Yano (Pharm. Inst., Univ. Kyushu, Japan). *Talanta*, 1959, **3** (1), 65–71.—The out-of-plane deformation vibrations of the aromatic ring of the tetralin series normally appear in almost the same frequency region as do those of the corresponding benzene series and, with a few exceptions, their frequencies are shifted in a regular manner according to substituents attached to the aromatic ring. All compounds that have an isolated CH group in the ring exhibit fairly strong absorption bands in the region round 800 cm^{-1}, which may be associated with the aromatic ring. Sixty-one compounds were investigated. G. P. COOK

1815. **Routine cryoscopic method for assaying β-picoline, γ-picoline and 2:6-lutidine.** K. A. Adey (Midland Tar Distillers Ltd., Four Ashes, nr. Wolverhampton, England). *Analyst*, 1959, **84**, 560–564.—The freezing-point apparatus is of the conventional type with an inner sample tube containing a stirrer and thermometer and an outer air jacket, the cooling liquid being contained in a Dewar jar. At the expected freezing-point the sample is seeded, and temp. readings (estimated to 0·01°) are made at 30-sec. intervals. The freezing-point corresponds to the first four consecutive readings at which the temp. remains constant. The liquid is then heated to 14° to 16°, a portion is withdrawn and its water content is determined immediately by the Karl Fischer method. Formulae are given for the calculation of the purity of the sample. Depressions of the freezing-point caused by the presence of certain other bases were found to be linear over the range examined (up to 10 moles %). The precision of the method is within 0·05° ($\simeq 0{\cdot}1\%$ purity). A. O. JONES

1816. **Analysis of natural gas samples by vapour-phase chromatography.** A. P. Hobbs. *Proc. 37th Ann. Conv., Nat. Gas. Ass. Amer.*, 1958, 33–36.—The gas is passed through two columns in series with an activated-charcoal trap between them. The first is a chromatographic column which separates the C_1 to C_6 paraffins but does not separate air from methane. The activated charcoal removes all hydrocarbons, and the second column, which is packed with molecular sieve type 5A, separates N, O and methane. The two columns have separate detectors and recorders. The peak areas obtained do not agree with the composition by vol. of the

samples. Empirical factors depend on the composition. Net B.Th.U. values calculated from vapour-phase chromatography and from distillation analysis are, however, in good agreement, and vapour-phase chromatography can therefore replace analytical distillation for routine control samples.
J. Inst. Petrol. Abstr.

1817. **Standard methods for the sampling and analysis of fuel gases.** British Standards Institution (2 Park Street, London, W.1). B.S. 3156: 1959, 140 pp.—The three sections describe well-established procedures for (*i*) taking snap and period samples of purified gases, storing the sample, and removing solid and liquid particles from the crude gases before entry into the sampling apparatus, specialised types of which are described; (*ii*) general analysis (CO_2, O, unsaturated hydrocarbons, H, CO, saturated hydrocarbons and N) by either the constant-vol. method or the soap-film method; the 'Metro' apparatus and method for the direct determination of N is also described; (*iii*) specific determinations, *viz*, of H_2S in crude gas (by pptn. as ZnS) and in purified gas (by the methylene blue and lead sulphide stain methods; total S in almost H_2S-free gas (by combustion, oxidation of SO_2 and titration of H_2SO_4); CS_2 (colorimetrically by reaction of its cryst. piperidine compound with Cu oleate), COS (colorimetrically as K_2S), thiols (from the decrease in concn. of Cu^{2+} after pptn. of mercaptides in standard Cu oleate soln.) and thiophen; condensable vapours (benzole); nitrogenous gum in purified gas (by impingement); tar-fog in crude gas (by impingement); NH_3 and naphthalene; HCN (by a volumetric and a colorimetric method); $Fe(CO)_5$, by decomposition in an ammoniacal soln. of methanol and H_2O_2, followed by colorimetric determination of Fe; water vapour and dew-point; sp. gr.; calorific value (by the Boys' Non-recording Calorimeter) and Aeration Test Burner number. Attention is drawn to some methods for determining oxides of N, and procedures are given for calculating the hydrocarbon-enrichment value, the Wobbe number, and the calorific value and sp. gr. (from the gas analysis). W. J. Baker

1818. **Testing mineral-oil hydrocarbons and similar products. Determination of the chlorine content. DIN 51577.** Fachausschuss Mineralöl- und Brennstoffnormung (Hamburg 1, Klosterwall 4, IV, City-Hof, Block B). *Erdöl u. Kohle*, 1959, **12** (7), 563–565.—The sample is burned and the gases are absorbed in alkali soln. The combustion may be carried out in a bomb with aq. Na_2CO_3 soln. as the absorbent, or in a stream of air in a quartz tube fitted with 2 absorbing tubes containing aq. NH_3 and aq. NaOH soln., respectively. The Cl^- content of the soln. obtained may be determined gravimetrically as AgCl or by the addition of excess of $AgNO_3$ and back-titration with NH_4SCN. This is a tentative standard. G. Burger

1819. **Determination of hydrogen sulphide in polysulphide hydrocarbon gases.** V. A. Garbalinskii (Odessa Petroleum Refining Works). *Zavod. Lab.*, 1959, **25** (6), 676–677.—Soln. of Cd or Zn acetate absorb unsaturated hydrocarbons as well as H_2S. The use of saturated $CuSO_4$ soln. in an Orsat apparatus is recommended for the selective absorption of H_2S. G. S. Smith

1820. **Trace analysis for total nitrogen in petroleum fractions. Adsorption - ter Meulen method.** E. C. Schluter, jun. (Union Oil Co. of Calif., Brea, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1576–1581.—The nitrogen compounds are concentrated from a relatively large vol. of hydrocarbon by adsorption on a silica gel column, which is then placed in a ter Meulen apparatus, where the nitrogen compounds and the hydrocarbons are desorbed from the silica gel by hydrogen and heat. The vapours pass through the Ni - Mg catalyst with little pyrolysis of the hydrocarbons. The nitrogen compounds are reduced to NH_3, which is collected and determined by conventional means. Recoveries from synthetic samples ranged from 85 to 110% at the 0·1 mg of N level. G. P. Cook

1821. **Arsenic in naphthas.** G. W. Powers, jun., R. L. Martin, F. J. Piehl and J. M. Griffin [Standard Oil Co. (Indiana), Whiting, Ind., U.S.A.]. *Anal. Chem.*, 1959, **31** (9), 1589–1593.—The As is separated from the naphtha by adsorption on silica gel impregnated with H_2SO_4. The adsorbent is then digested with H_2SO_4, HNO_3 and $HClO_4$ to remove organic matter and dissolve the As, which is subsequently converted into AsH_3 and determined spectrophotometrically at 540 mμ with Ag diethyldithiocarbamate in pyridine. The precision and accuracy are about ±3% relative or 0·3 part per thousand million, whichever is greater. G. P. Cook

1822. **Determination of microgram quantities of arsenic in naphthas with oxy-hydrogen burner.** D. K. Albert and L. Granatelli (American Oil Co., Texas City, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1593–1596.—The sample is burned in the flame of a oxy-hydrogen burner and the combustion products are absorbed in NaOH soln. The As in this soln. is determined spectrophotometrically at 540 mμ with Ag diethyldithiocarbamate in pyridine. The standard deviation varies from 1 to 8 parts per thousand million at the 8 to 78 parts per thousand million levels, respectively. The relative error at the corresponding levels varies from 12 to 6%, respectively. The limiting concn. is 2 parts per thousand million. G. P. Cook

1823. **Modification of Mott's method for the determination of pyritic sulphur in coal.** W. van Hees and E. Early (Baltimore Gas and Electric Co., Baltimore, Md., U.S.A.). *Fuel, London*, 1959, **38** (4), 425–428.—In Mott's method two samples are extracted, one with 5 *N* HCl and the other with 2 *N* HNO_3. The pyritic sulphur content is calculated from the difference in the iron content of the extracts. The modification consists in extracting the same sample with HCl and HNO_3 successively. The pyritic sulphur content is calculated from the iron content of the second extract. The modified method is as precise as Mott's method and quicker, but does not permit the simultaneous determination of SO_4^{2-}. G. Burger

1824. **Determination of menthyl salicylates in lemon oil.** W. L. Stanley (Fruit and Veg. Chem. Lab., Agric. Res. Service, U.S. Dept. of Agric., Pasadena, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 643–646.—Menthyl and *m*-homomenthyl salicylates, which if added to artificial lemon oil may render it spectrally indistinguishable from genuine lemon oil, each have an R_F value of 0·92 when tested on chromatostrips by ascending development with diethyl ether - light petroleum, whereas the coumarins and psoralens, responsible for the spectral absorption of lemon oil at 315 mμ, have R_F values $< 0·50$. By the procedure described,

the menthyl salicylates are separated chromatographically on paper coated with silicic acid by descending development with the diethyl ether - light petroleum mixture, the appropriate section detected in u.v. light is removed, and the u.v. absorption of its ethanol extract is determined at 307 mμ. A. A. ELDRIDGE

1825. **Determination of active oxygen in cosmetic emulsions containing zinc peroxide.** J. Pokorný, M. Karvánek and V. Pokorná (Inst. Chem. Technol., Prague). *Prûmysl Potravin*, 1959, **10** (7), 385–386.—Various methods were compared and iodimetric titration and colorimetric determination were found to be the most suitable. *Iodimetric method*—To the sample (10 g) add $CHCl_3$ (20 ml), H_2SO_4 (20%) (20 ml) and acetic acid (50%) (10 ml), shake, transfer to a separating-funnel and allow the layers to separate. Filter the aq. layer into a 100-ml flask, wash the funnel and filter twice with H_2O and dilute the combined filtrate and washings to vol. To an aliquot (10 ml) add KI soln. (15%) (20 ml) and 20% H_2SO_4, set aside for 15 min. in the dark and titrate with 0·1 *N* $Na_2S_2O_3$, with starch soln. as indicator. *Colorimetric determination*—To the sample (0·05 to 0·1 g) in a separating-funnel add benzene (10 ml), acetic acid (50%) (5 ml) and H_2SO_4 (20%) (10 ml), shake, add H_2O (20 ml) and filter the lower layer into a 100-ml flask; wash the funnel and the filter with H_2O, add 5 ml of reagent (150 g of $TiCl_4$ dissolved in 1·5 litres of 20% HCl and diluted with 1 litre of H_2O) and dilute to vol. Measure the yellow colour photometrically (blue filter) and compare with a calibration curve. J. ZÝKA

1826. **Identification of *p*-phenylenediamine in hair dye.** T. Brouwer (Keuringsdienst van Waren, 's-Gravenhage, Netherlands). *Chem. Weekbl.*, 1959, **55** (26), 325–327.—A sensitive and specific method for the identification of *p*-phenylenediamine (**I**) in small samples of hair dye is described. *Procedure*—Mix 0·05 ml or 0·05 g of the sample with 1 to 2 drops of conc. HCl and evaporate the excess of HCl on a steam bath. Mix the residue with 1 drop of H_2O and sufficient anhyd. Na_2CO_3 to produce a dry substance. Mix this with ≃1 ml of diethyl ether and decant through a filter of ≃5 cm diam., and repeat the extraction until 2·5 ml of filtrate is obtained. In this soln. **I** is identified as follows—(*i*) *Indamine reaction*—Evaporate 0·5 ml of the ether soln. to dryness, add 2 drops of aniline soln. (1 drop of aniline in 50 ml of 6% acetic acid) and a few crystals of $K_2S_2O_8$ and mix. If **I** is present a blue colour is produced. (*ii*) *Reaction with vanillin and HCl*—To 0·5 ml of the ether soln. add 1 drop of vanillin soln. (1 g of vanillin in 100 ml of diethyl ether) and a trace of conc. HCl. When the soln. has evaporated to dryness, wash the dry residue with ether. If the residue turns violet after moistening with water, **I** is present. (*iii*) *Chromatographic method*—Apply a small drop of conc. HCl to a filter-paper so that a spot of about 0·5 cm diameter is formed. Pipette slowly 0·1 ml of the ether soln. on this spot and allow to dry. Add 1 drop of vanillin soln. to the spot and from the colour intensity observed determine the amount of ether soln. which must be taken for the following chromatographic method. Mix 35 ml of *n*-butanol, 10 ml of conc. HCl and 20 ml of H_2O in a separating-funnel. Leave the mixture overnight and use the upper layer as the eluent. Equilibrate the filter-paper (Whatman No. 1) in the vapour of this mixture for some hours, then apply the soln. to be analysed and also 2·5 μg of **I** to the starting line. Use ascending chromatography for 24 hr., allow the paper to dry and spray with vanillin soln. Compare the R_F values and colours of the spots to identify **I** in the test soln. M. J. MAURICE

1827. **X-ray powder diffraction data of azoic coupling components.** I. Schnopper, J. O. Broussard and C. K. LaForgia (U.S. Customs Lab., New York, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1542–1549.—Data are presented for 15 organic compounds. These permit the identification of the azoic coupling components of Naphthol AS (C.I. Azoic Coupling Component 2), some of its derivatives and related compounds. K. A. PROCTOR

1828. **Ascorbimetric determination of resazurin and resorufin and their derivatives.** E. Ružička (Palacký Univ., Olomouc, Czechoslovakia). *Z. anal. Chem.*, 1959, **168** (6), 414–417 (in German).—Resazurin and resorufin and their derivatives may be determined by potentiometric titration with ascorbic acid to give colourless or blue - green leuco compounds. An ethanolic soln. of the compound is placed in a titration cell with an equal volume of 4 *N* HCl under a continuous flow of CO_2, and the mixture is heated to 65°. Titration is then carried out with 10^{-3} *M* ascorbic acid, with a platinum electrode *vs.* a S.C.E. The determination can be carried out in the presence of gallocyanine and azo dyes, but aminophenoxazinones, oxazines and thionine interfere. S. M. MARSH

1829. **Determination of starch solubilised by means of irradiation.** J. Trzebiński and L. Ehrenberg (Inst. of Org. Chem. and Biochem., Univ., Stockholm, Sweden). *Acta Chem. Scand.*, 1959, **13** (6), 1075–1080.—Satisfactory aq. extraction of starch by boiling for 30 min. is effected if the vegetable material is extracted with boiling ethanol and then irradiated with ≮500,000 rad of γ-radiation (^{60}Co source or Van de Graaf accelerator) and ground in a ball mill. Doses of radiation up to $1·6 \times 10^6$ rad do not affect the iodine colorimetry, but doses of 10×10^6 rad shift the absorption max. and so lower the extinction at 590 mμ. Irradiation of dried samples that had not been extracted with ethanol did not lead to increased solubilisation of the starch. E. J. H. BIRCH

1830. **Rapid photometric determination of nitrogen in nitrocellulose.** Tetsuro Murakami and Eizen Ishii (Kogakuin, Kashiwagi, Shinjuku-ku, Tokyo). *Japan Analyst*, 1959, **8** (4), 257–259.—Swann's method (*Anal. Abstr.*, 1957, **4**, 871) was modified to be applicable to those samples insol. in the reagent. Such a sample (10 mg) is dissolved in H_2SO_4 (95%) (5 ml) and mixed with the reagent (5 g of $FeSO_4.7H_2O$ in 250 ml of water, mixed with 750 ml of conc. H_2SO_4) after 10 to 15 min. The extinction is measured at 517 mμ after shaking for 5 min. K. SAITO

1831. **Accelerated analysis of cotton - viscose rayon blends with formic acid - zinc chloride reagent.** F. Brunello and G. Rossi. *Tinctoria*, 1959, **56** (7), 290–291.—The Schlütter - Marschal method with formic acid - zinc chloride is accelerated by working at 55° rather than at 40°, thus reducing the time required from 2·5 hr. to 1 hr. A correction factor of 0·965 is applied to allow for the probable formation of small amounts of formylcellulose. B.C.I.R.A. ABSTR.

1832. **A non-hydrolytic spectrophotometric method for determining lignin in high-purity pulps.** R. Bartunek (Vereinigte Glanzstoff-Fabriken A.-G., Oberbruch, Germany). *TAPPI*, 1959, **42**, 553–556.—"Lignin number" is generally expressed as a function of the intensity of the yellow colour developed when wood pulp is treated with 13% HNO_3 at 40°. Ultra-violet spectrophotometric investigations on HNO_3 extracts from various pulps showed that extinction maxima at 261 mμ and 330 to 338 mμ were pronounced enough to allow differentiation. The sample pulp (15 g, calculated on a moisture-free basis) was cut into 5-mm squares and placed in a glass-stoppered flask containing 100 ml of 13% HNO_3 at 40°. After 45 min. at this temp. the mixture was filtered through sintered glass, the yellow filtrate was cooled rapidly and immediately examined with a Beckman DU spectrophotometer, with a hydrogen source below 325 mμ and a tungsten lamp above 325 mμ. Extinction values are given for different types of pulp (spruce, birch, western hemlock, chestnut, straw, sulphite, sulphate) and for an extract of Björkman lignin prepared by extraction of woodmeal with aq. dioxan at 20°.
R. AICHER

1833. **Detection of preservatives in wood with catechol violet.** W. J. Wilson (Forest Res. Inst., P. B. Whakarewarewa, Rotorua, N. Zealand). *Anal. Chim. Acta*, 1959, **21** (2), 150–154.—The application to wood of catechol violet soln. (0·1% in 5% ammonium acetate soln.) produces a blue colour in the presence of the common wood preservatives (containing Cr, Cu and sometimes Zn, As or F). A second reagent soln. (0·05% of catechol violet and 0·05% of $ZrOCl_2$ in 0·1 *N* HCl) gives a pink colour in the presence of As or F, remaining blue in their absence. T. R. ANDREW

1834. **Determination of monomeric vinyl acetate.** J. Dodomka (Res. Inst. Macromol. Chem., Brno, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (7), 363–365.—The method based on the addition of bromine was improved by using a stable soln. of bromine in glacial acetic acid. *Procedure*—Into a dry 100-ml flask place acetic acid soln. (50%) (50 ml) and the sample of monomeric vinyl acetate (0·4 ml). Dilute with acetic acid to vol., mix, and transfer an aliquot (10 ml) to a flask containing H_2O (100 ml). Titrate with 0·1 *N* Br (2·7 ml of Br in 1 litre of glacial acetic acid) till slightly yellow; add 0·5 to 1 ml of the reagent in excess, and 10% KI soln. (10 ml) and titrate with 0·1 *N* $Na_2S_2O_3$ till colourless. When determining monomeric vinyl acetate in an emulsion of poly(vinyl acetate), $CHCl_3$ must be added before the addition of KI soln. to prevent absorption of liberated iodine by the emulsion. J. ZÝKA

1835. **Bromatometric determination of the mean functionality of higher phenolic fractions.** V. Vorobjov (Inst. of Insulators, Bratislava, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (7), 384–387.—Bromatometric titration with amperometric indication is a suitable industrial analytical method for the determination of the mean functionality of higher phenolic fractions from coal tar, used in the production of resins. *Procedure*—Dissolve the sample (0·4 to 0·6 g) in H_2O (500 ml); to 50 ml add KBr (1 g), conc. HCl (5 ml) and methanol (50 ml). Titrate amperometrically with 0·1 *N* $KBrO_3$, with a rotating platinum indicator electrode and a S.C.E., and apply a blank correction. J. ZÝKA

1836. **Analytical control of polyester condensation.** W. Reiser. *Farbe u. Lack*, 1959, **65** (7), 370–373.—The esterification of a dibasic acid anhydride and a polyhydric alcohol to form a polyester may be followed by determinations of the acid values in anhydrous and aq. media. The former determination is carried out by dissolving the resin in anhyd. methanol - benzene (1:2) and titrating with 0·1 *N* methanolic KOH. For the latter determination, the resin is dissolved in ethyl acetate - butanol - water (15:7:3), heated for 30 min. at 75° and titrated with 0·1 *N* aq. KOH. The difference between the two acid values gives a measure of the anhydride groups present, and, together with the sap. value, enables the content of anhydride, mono-ester and di-ester to be calculated.
L. A. O'NEILL

1837. **Analysis of terephthalic polyester wire-lacquers.** D. Hummel (Cologne Univ., Germany). *Farbe u. Lack*, 1959, **65** (8), 440–449.—A scheme for the analysis of these lacquers has been developed. The non-volatile content of the lacquer is determined by heating, and the ash is obtained and analysed spectroscopically. The medium is separated from the solvent by pptn. with diethyl ether and its sap. val. and mol. wt. (cryoscopic in phenol) are determined and the infra-red spectra examined. It is then saponified and the nature of the alcohol component (glycol or glycerol) is established by gas chromatography and infra-red spectroscopy. The solvent is obtained by distillation, after the pptn. of the medium, and the phenol content is determined by extraction with strong alkali. The nature of the phenolic components is examined by gas chromatography and infra-red spectroscopy and the non-phenolic solvents by the latter technique. The application of the scheme to two typical lacquers is described. L. A. O'NEILL

1838. **Determination of the non-volatile content of urea resins.** P. R. Deeleman and A. C. Van Der Schee. *Verfkronick*, 1959, **32** (6), 261–265.—The effects of time and temp. of heating, and of film thickness, on the determination of the non-volatile content have been studied with urea resins of different degrees of butylation. The non-volatile content, when determined on mixtures of the urea resin with an alkyd resin, is always lower than for the urea resin alone, and varies with the acid and hydroxyl content of the alkyd resin.
L. A. O'NEILL

1839. **Polarographic determination of accelerators, anti-ageing agents and other organic substances used in the rubber industry. II.** F. Mocker (Veith-Gummiwerke A.-G., Höchst, Odenwald, Germany). *Kautsch. u. Gummi*, 1959, **12** (6), 155–159.—N-*cyclo*Hexylbenzothiazole-2-sulphenamide and benzothiazol-2-yl diethyldithiocarbamate are extracted from raw rubber with $CHCl_3$ - methanol (1:2) and determined by polarography at pH 5·4 and 7·8, respectively (*cf. Ibid.*, 1958, **11**, 281). In vulcanised rubber the compounds can only be determined indirectly since decomposition to 2-mercaptobenzothiazole occurs during vulcanisation. Samples containing zinc oxide are extracted with acetic acid and the zinc is removed by pptn. with H_2S before polarography. The precision of the procedure is $\simeq \pm 2\%$. W. T. CARTER

1840. **Polarographic determination of pentaerythritol trinitrate in the presence of nitroglycerin.**

W. M. Ayres and G. W. Leonard (Research Dept., U.S. Naval Ordnance Test Station, China Lake, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1485–1487.—Pentaerythritol trinitrate can be determined rapidly by the method described with an accuracy of about ±1%. Interference due to heavy-metal salts is negligible, but the presence of some organic nitrates would necessitate the application of a correction. K. A. PROCTOR

See also Abstracts—**1638**, Spot reagents for functional groups. **1639**, Trace metals in dyestuff intermediates. **1687**, Determination of B in org. compounds. **1705**, Determination of ammonium salts. **1707**, Nitrate in explosives. **1710**, Phosphate in org. compounds. **1853**, Analysis of plant waxes. **1864**, Determination of mercapto groups. **1908**, Determination of piperazine. **1923**, Formic acid and formates in foods. **1971**, Org. mercury compounds in agricultural chemicals. **2029**, Analysis of propylene polymers, alkylbenzenes and waxes.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1841. **The wet oxidation of bone. Digestion with 100 per cent. sulphuric acid followed by the addition of dioxonium perchlorate [73·6% perchloric acid].** G. F. Smith and H. Diehl (Noyes Chem. Lab., Univ. of Illinois, Urbana, U.S.A.). *Talanta*, 1959, **3** (1), 41–46.—The sample is first oxidised with 100% H_2SO_4 (prep. described) at 300° to 325°, then with 73·6% $HClO_4$ (Smith and Koch, *Ind. Eng. Chem., Anal. Ed.*, 1931, **3**, 52), with 1 mg of vanadium catalyst, at 200° to 210°. G. P. COOK

1842. **Complexometric determination of sodium in blood serum.** M. Dugandžic, H. Flaschka and A. Holasek (Med.-Chem. Inst. d. Univ., Graz, Austria). *Clin. Chim. Acta*, 1959, **4** (6), 819–822.—Sodium is ppt. from a trichloroacetic acid filtrate of the serum as sodium zinc uranyl acetate. The ppt. is filtered off, washed with ethanol and re-dissolved, and the Zn is titrated with EDTA soln., with dithizone as indicator.

H. F. W. KIRKPATRICK

1843. **Determination of calcium in plant material by atomic-absorption spectrophotometry.** D. J. David (C.S.I.R.O., Div. of Plant Industry, Canberra, A.C.T., Australia). *Analyst*, 1959, **84**, 536–545.—The apparatus used is that previously described (*Ibid.*, 1958, **83**, 655) with the addition of one lens between the hollow-cathode tube and the Lundegårdh flame and another between the flame and the spectrograph slit. The sample is wet-ashed with H_2SO_4, $HClO_4$ and HNO_3, excess of $HClO_4$ and HNO_3 are removed by heating, and ppt. SiO_2 by filtration after dilution. To an aliquot of the filtrate and to the standard soln. are added soln. providing specified amounts of the chlorides of Mg, Na and K. Sample and standard soln. are then subjected to spectrographic analysis as previously described (*loc. cit.*). Thorough dehydration of SiO_2 and thorough washing of the insol. residue are important. A. O. JONES

1844. **Determination of strontium in biological materials.** H. Holeyšovská-Kozáková (Inst. Hyg. and Industrial Diseases, Prague). *Pracovní Lekařství*, 1959, **11** (6), 311–315.—Strontium can be ppt. with rhodizonate and determined flame-photometrically. *Procedure*—Adjust the urine (4 ml) with dil. HCl to pH 4, and add a satd. soln. of Na rhodizonate (1 to 2 ml) and ethanol (4 ml). Set aside for 2 to 3 hr., separate the ppt. in a centrifuge and reject the supernatant liquid; wash the ppt. with a mixture of H_2O and ethanol (2:1) (3 ml) and centrifuge. Reject the supernatant liquid, dissolve the ppt. in HCl (3·7%) (0·2 ml), add a soln. of LiCl (2·5 ml ≡ 1 mg of Li) as internal standard, dilute to 10 ml and measure the extinction at 461 mμ. The method is suitable for determining 50 to 200 μg of Sr per ml with an average error of ±6%. A large excess of phosphate can be separated by means of the anion-exchange resin Amberlite IR-4B. J. ZÝKA

1845. **Detection and urinary elimination of phenaglycodol.** L. Cima and F. Fassina (Ist. Farmac., Univ. Padova, Italy). *Farmaco, Ed. Prat.*, 1959, **14** (7), 416–422.—Phenaglycodol (2-*p*-chlorophenyl-3-methylbutane-2:3-diol) is extracted with ether from urine made alkaline with NaOH soln. After removal of the ether the residue is treated with the following reagent. To 400 ml of 10% NaOH soln. add 200 ml of H_2O and 60 g of molybdic acid. Boil for 30 min., cool and add 250 ml of phosphoric acid (d = 1·71). Cool and make up to 1 litre. After heating the residue with the reagent on a water bath for 20 min., the blue colour is read at 810 mμ. The Beer - Lambert law is obeyed in the range from 0·5 to 20 μg per ml. L. ZANONI

1846. **Quantitative determination of oleandomycin in bile and urine.** G. Gerner and W. Fischer (Med. Universitätsklinik, Erlangen). *Arzneimittel-Forsch.*, 1959, **9** (6), 354–356.—The oleandomycin (**I**) content of bile and urine samples was determined by comparison of the zones of inhibition of a culture of vegetative cells of *Bacillus cereus* var. *mycoides* (ATCC 9634) with those produced by standard soln. of **I**. From 0·2 μg to 100 μg of **I** per ml can be determined with a mean coeff. of variation of ±9%. A. G. COOPER

1847. **Determination of pentoses in plasma by Bial's reaction.** E. Scala, M. Beati and G. Ruiz (Ist. di Chim. Biol., Univ. di Napoli). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (13), 756–760.—To determine pentoses in materials containing hexoses (*e.g.*, plasma), Dische's modification of Bial's orcinol reaction (*Amer. J. Ophthalmol.*, 1953, **36**, 54) proved the simplest and most reliable method. It depends on the difference between the extinctions at 670 mμ and 580 mμ. This difference is a function only of the concn. of pentose, which is then determined by reference to curves previously obtained by submitting mixtures of pentose (0 to 30 μg) and glucose (50, 100 and 150 μg) of known concn. to the procedure. Pentose (2 to 30 μg) was determined in samples (1 ml) of deproteinised plasma containing ≯150 μg of glucose, and, within these limits, the recovery of pentose added to serum was complete. By the method described, the free pentose content of serum varied from 8 to 30 μg per ml. C. RAINBOW

1848. **Sensitive colorimetric method for the estimation of 2-deoxy sugars with the use of the malonaldehyde - thiobarbituric acid reaction.** V. S.

Waravdekar and L. D. Saslaw (Biochem. Branch, Armed Forces Inst. of Path., Washington, D.C., U.S.A.). *J. Biol. Chem.*, 1959, **234** (8), 1945–1950.—The 2-deoxy sugars are oxidised with HIO_4 and the resulting malonaldehyde is condensed with thiobarbituric acid to give a pink chromogen, which is determined spectrophotometrically at 532 mμ. The specificity of the method is increased if alkali is added after the colour development. This causes the absorption max. to shift to the region of 557 mμ; the extent of the shift depends on the amount of alkali used. J. N. ASHLEY

1849. **Polysaccharide analysis of liver biopsy specimens obtained at laparotomy.** S. A. Barker, M. Stacey and D. J. Tipper (Chem. Dept., The Univ., Edgbaston, Birmingham, England). *Clin. Chim. Acta*, 1959, **4** (6), 861–866.—A general method of fractionation, with cetrimide, for use with biopsy specimens of about 1 g (wet wt.) is described. The method is designed for conditions involving metabolic disturbance where abnormal storage of a polysaccharide constituent occurs. H. F. W. KIRKPATRICK

1850. **The determination of α-keto acids in cerebrospinal fluid.** E. Zelníček (Inst. Med. Chem., Med. Fac., Univ. Brno, Czechoslovakia). *Čas. Lék. Čes.*, 1959, **98** (27), 856–859.—α-Keto acids are converted into 2:4-dinitrophenylhydrazones and, after extraction, separated chromatographically on Whatman No. 1 paper, with *n*-butanol - ethanol (96%) - aq. NH_3 (1%) (6:1:3) as solvent, or electrophoretically on Whatman No. 1 paper in 0·05 *M* $NaHCO_3$ at 400 mV and 10 to 18 mA. After the separation and elution with *N* $NaHCO_3$, the yellow colour is measured at 380 mμ; 0·22 to 0·72 mg of pyruvic acid per 100 ml can be thus determined. J. ZÝKA

1851. **Quantitative determination of carotene by paper chromatography.** A. Jensen (Norwegian Inst. of Seaweed Res., N.T.H., Trondheim, Norway). *Acta Chem. Scand.*, 1959, **13** (6), 1259–1260 (in English).—Carotene in dried seaweed and grass is determined after separation on a kieselguhr-loaded paper. *Procedure*—The ground sample (1 g) is moistened with 2 ml of water and after 15 min. is extracted in the dark with successive portions of 25 ml of acetone under N until the extract is colourless. The extract is evaporated and made up to a known vol. (10 or 25 ml) with acetone. An aliquot (0·2 to 0·8 ml) is applied to the centre of a kieselguhr-loaded circular paper (S. & S. No. 287) with continuous evaporation of the acetone with a stream of N. The chromatogram is then run by Rutter's technique (*Nature*, 1948, **161**, 435) with light petroleum (boiling-range 60° to 80°), and as soon as the carotene zone has separated (close to the solvent front) the development is interrupted and the carotene zone is cut out and extracted, in a small tube with a capillary end, with 5 ml of acetone. The extinction at 456 mμ is determined and the carotene content is calculated from the extinction coeff. of β-carotene in acetone ($E^{1\%}_{1cm}$ = 2490). Results are slightly higher than those by column chromatography. E. J. H. BIRCH

1852. **Rapid method of total lipid extraction and purification.** E. G. Bligh and W. J. Dyer (Fisheries Res. Board of Canada, Technol. Sta., Halifax, Nova Scotia). *Canad. J. Biochem. Physiol.*, 1959, **37** (8), 911–917.—The wet tissue is homogenised in a Waring Blendor with a mixture of $CHCl_3$ and methanol in such proportions that a miscible system is formed with the water in the tissue. Dilution with $CHCl_3$ and H_2O causes the homogenate to separate into two layers, the $CHCl_3$ layer containing all the lipids practically free from non-lipids, and the methanol - H_2O layer containing the non-lipids. The volumes of $CHCl_3$, methanol and water before and after dilution must be kept in the proportions 1:2:0·8 and 2:2:1·8, respectively. An aliquot of the $CHCl_3$ extract is evaporated to dryness and the residue weighed. N. E.

1853. **Analysis of plant waxes by means of chromatography and X-ray diffraction.** N. Wiedenhof (Lab. for Gen. and Tech. Biol., Tech. Univ., Delft, Holland). *J. Amer. Oil Chem. Soc.*, 1959, **36** (7), 297–300.—Candelilla, carnauba, sugar-cane and Java kapok waxes have been resolved into their constituents by these techniques. For chromatography, the wax (0·1 to 1 g) is dissolved in 20 ml of hot *n*-heptane and applied to a column (24 cm × 2·4 cm) of Al_2O_3 (Woelm, anionotropic, pH 4, Grade I anhyd.), the top of which is heated by an i.r. lamp. The column is then successively eluted with *n*-heptane, *n*-heptane containing 1 to 5% of diethyl ether, *n*-heptane containing 1 to 5% of propanol, and *n*-heptane containing 1 to 5% of acetic acid. Recoveries of 90% of the wax are obtained, and the fractions are identified by their sap. and acid values. X-ray diffraction diagrams are obtained with the Cu-K_α radiation (30 to 35 kV, 300 to 350 mA) from flat powdered specimens 0·5 mm thick. G. R. WHALLEY

1854. **Comparative studies on the reproducibility and sensitivity of various methods of determination of choline.** L. Acker and H. Kaiser (Univ.-inst. f. Lebensmittelchem., Frankfurt/Main). *Pharm. Zentralh.*, 1959, **98** (7), 347–356.—The methods of Roman (*Biochem. Z.*, 1930, **219**, 218), Appleton *et al.* (*Anal. Abstr.*, 1954, **1**, 758), Kushner (*Anal. Abstr.*, 1957, **4**, 226) and Kates (*Canad. J. Biochem. Physiol.*, 1954, **32**, 571) are compared. The reineckate absorptiometric method of Kates is the most precise, but it needs a high concn. of choline and takes 3 hr. for completion. For a series of analyses, the choline ennea-iodide titrimetric method of Roman is preferred, because it is more rapid and almost as precise. A. R. ROGERS

1855. **The determination of α-amino nitrogen by the copper method.** E. Bottini, P. Strigini and G. Antognoni (Clin. Pediatr., Univ. Roma). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (13), 772–775.—The determination of α-amino groups by the copper method (Pope and Stevens, *Biochem. J.*, 1939, **33**, 1070) and its modifications depends on the formation of soluble amino acid - copper compounds. As applied to urine, values obtained by the method were reproducible, but higher than those obtained by Van Slyke's method. Soluble copper compounds, titratable like amino acids, were also formed by simple peptides and by citric, pyruvic, tartaric, ascorbic and salicylic acids. By plotting μ-equiv. of thiosulphate consumed in titrating the copper derivatives against μmoles of test substance (*S*), four types of reaction of *S* with Cu were recognisable—(*i*) 1*S*:1Cu (*e.g.*, glycylglycine, leucylglycine, glycylglycylglycine); (*ii*) 2*S*:1Cu (*e.g.*, alanine, glycine, glutamic acid); (*iii*) 4*S*:3Cu (*e.g.*, histidine, citrate, tartrate); and (*iv*), a type of reaction not easily interpreted (*e.g.*, salicylate, ascorbate, pyruvate). Preliminary tests, involving

pure substances dissolved in water and in urine, indicated that the separation of the copper derivatives on ion-exchange columns was possible, those of the above amino acids and peptides being eluted from Dowex 50 at pH 13 after elution of the carboxylic acids (except salicylic) at pH 1.

C. RAINBOW

1856. **Critical evaluation of methods used in aminoaciduria investigation.** J. Opieńska-Blauth (Biochem. Dept., Med. Acad., Lublin, Poland). *Clin. Chim. Acta*, 1959, **4** (6), 841–860 (in English).—Various aspects of the chromatographic and other methods used in assessing aminoaciduria are discussed and illustrated experimentally. A recommended procedure is presented.

H. F. W. KIRKPATRICK

1857. **Ion-exchange chromatography of amino acids. Semi-automatic method of operation with cationic exchange resin columns.** P. B. Hamilton and R. A. Anderson (Alfred I. du Pont Inst., Nemours Foundation, Wilmington, Del., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1504–1512.—Amino acids found in protein hydrolysates were separated chromatographically by using a single column (0·636 cm × 100 cm) packed with 40-μ particles of an 8·5% cross-linked sulphonated polystyrene cationic exchange resin. The fraction collector automatically controls the changes of temp. and buffer soln. after collection of pre-arranged fractions. All fractions are analysed colorimetrically with ninhydrin. The sharp peaks obtained give good resolution and recovery of the amino acids. The apparatus and typical chromatograms are illustrated.

G. P. COOK

1858. **Polarographic determination of amino acids by complex formation with copper.** C. Edon. *Compt. Rend.*, 1959, **249** (3), 416–417.—The complex CuR_2 is formed by progressive addition of $Cu(NO_3)_2$ soln. to a soln. of the amino acid in phosphate buffer soln. (pH 7·52). Salicylaldehyde in dimethylformamide is added to form a complex with the excess of Cu and to act as a polarographic indicator.

H. F. W. KIRKPATRICK

1859. **Quantitative determination of amino acids by combined displacement and paper chromatography.** F. Lundquist (Univ. Inst. of Forens. Med., Copenhagen, Denmark). *Clin. Chim. Acta*, 1959, **4** (6), 784–787 (in English).—The material (urine, plasma, tissue or protein hydrolysate) is desalted by passage through a small column of Dowex 50 (H^+ form). The adsorbed amino acids are eluted with aq. NH_3 and are separated into four groups by means of a column of Dowex 50-X4 (H^+ form), pretreated with *iso*propylpyrazole, pyrazole, nicotinamide and aq. NH_3, and the use of a fraction collector. A two-dimensional paper chromatogram is run on each of the four groups. By this method all amino acids are clearly separated and may be determined by the ninhydrin reaction.

H. F. W. KIRKPATRICK

1860. **Collaborative test of Moore and Stein's resin-chromatographic method for determining amino acids.** A. E. Bender, J. A. Palgrave and B. H. Doell (Res. Dept., Bovril Ltd., 148 Old St., London). *Analyst*, 1959, **84**, 526–536.—The collaborative assays were carried out in ten laboratories. Nine amino acids, *viz*, glycine, phenylalanine, lysine, valine, serine, threonine, aspartic acid, glutamic acid and tyrosine, were recovered with a mean recovery of 100 ± 2%. Alanine, leucine, *iso*leucine, arginine, histidine, proline and methionine were determined with an error of ± 8%. The determination of cystine is still not satisfactory, but a correction factor can be applied. Accuracy depends on a close attention to detail. The results of the assays are tabulated and discussed.

A. O. JONES

1861. **Paper electrochromatography of amino acids of blood serum.** V. Jirgl (Inst. for Haematology and Blood Transfusion, Prague). *Experientia*, 1959, **15** (6), 235–237 (in German).—This method combines the techniques of electrophoresis and chromatography. The free amino acids of serum are separated into neutral, acid and basic components on Whatman No. 3 paper, with a paper-electrophoresis apparatus at 210 V and 14 mA in pyridine - acetic acid - water (1:1:45) (pH 4·7). Separation of these fractions in the second direction is effected by means of the descending technique with *n*-butanol - acetic acid - water (4:1:5) on a Whatman No. 3 paper sewn to the electropherogram.

P. NICHOLLS

1862. **Factors affecting the ultra-micro determination of tryptophan, kynurenine and their derivatives.** A. A. Hakim (Biochem. Res. Lab., Miami Heart Inst., Miami Beach, Fla., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (2), 170–176.—The resolution of DL-tryptophan and kynurenine by paper chromatography and electrophoresis has been studied. It is postulated that the separation of D- and L-tryptophan on paper is due to adsorption on the optically active surface of the paper, such activity being induced by the methanol used as solvent. The electrolyte used for the electrophoresis influenced the mobility, separation and u.v. spectra. Pretreatment of the paper for chromatography did not affect the R_F values, but interfered with the quant. determination. The elution was studied as a function of pH, and it was found that sterile distilled water or a $NaOH$ - $NaHCO_3$ soln. (concn. not stated) was the best eluent. The u.v. spectra of D- and L-tryptophan were altered by continued u.v. irradiation, owing to the formation of degradation products.

T. R. ANDREW

1863. **Sensitive colour reaction for the paper-chromatographic detection of iodide, iodinated tyrosines and thyronines.** R. Gmelin and A. I. Virtanen (Found. for Chem. Res., Biochem. Inst., Helsinki, Finland). *Acta Chem. Scand.*, 1959, **13** (7), 1469–1470 (in English).—The reaction depends on the catalysis by traces of I^- of the reduction of a $Fe(CN)_6^{3-}$ - Fe^{3+} mixture by $NaAsO_2$. *Procedure*—The reagent is made up as three stock soln., (*A*) 2·7 g of $FeCl_3.6H_2O$ in 100 ml of 2 *N* HCl, (*B*) 3·5 g of $K_3Fe(CN)_6$ in 100 ml of H_2O, (*C*) 5 g of $NaAsO_2$ in 30 ml of cooled *N* NaOH to which is added 65 ml of 2 *N* HCl with vigorous stirring. The stock soln. are mixed (5:5:1) immediately before use and kept in the dark. The mixed reagent is sprayed in a dim light on to both sides of the dried chromatogram, which is put between glass plates without pressure. After 15 min., blue spots develop and the chromatogram is washed and dried. The spots may be determined photometrically (filter FE 54). The limit of detection is 0·001 μg for iodide and 0·002 μg for thyroxine, tri-iodothyronine and di-iodotyrosine. Some reducing agents also give blue spots, but only in quantities > 1 μg per spot.

E. J. H. BIRCH

1864. **Mercurimetric determination of mercapto groups in skin homogenates by the rotating platinum**

electrode. A. Montagnani, P. Santoianni and F. di Ieso (Univ. di Napoli, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (14), 903–905.—The method, modified from that of Kolthoff *et al.* (*Anal. Abstr.*, 1954, **1**, 1326), depends on the specific reaction involving one Hg^{2+} ion and one -SH group. Samples, prepared at 0°, consisted of finely ground fresh rabbit epiderm, homogenised in 10 ml of $1{\cdot}5 \times 10^{-3}$ *M* EDTA (disodium salt) per 100 mg of tissue, and then centrifuged. Total -SH was determined on one portion of the supernatant liquid. A second portion, after deproteinisation by addition of an equal volume of 2·5% sulphosalicylic acid, served for the determination of non-protein -SH. To quantities corresponding to 2 mg of fresh tissue was added a soln. (30 ml) containing NaH_2PO_4 (0·01 *M*), Na_2HPO_4 (0·08 *M*) and KCl (0·5 *M*) at pH 7·3. The rotating platinum electrode was placed in this soln. and the cell so formed was connected to a reference calomel electrode by a KCl bridge. Titrations were carried out with 10^{-3} *M* $HgCl_2$ added in portions of 0·1 ml at intervals of 1 min., the end-point being indicated galvanometrically. The presence of oxygen did not affect the results and the precision of the method was high. Only small differences were found between the total and non-protein -SH contents (respectively 2·40 and 2·15 mg per 100 mg of tissue). Presumably this reflected the low solubility of epidermal protein.

C. RAINBOW

1865. **Determination of cystine with *p*-aminodimethylaniline.** E. A. Robinson (Arthur R. Davis & Co. Ltd., 74 Borough High St., London). *Anal. Chim. Acta*, 1959, **21** (2), 190–192.—The determination of cystine by reduction to cysteine and measurement of the blue colour formed with *p*-aminodimethylaniline (**I**) in the presence of Fe^{3+} (*cf.* Mecham, *J. Biol. Chem.*, 1943, **151**, 643) has been critically re-examined and improved. The max. colour development occurs at a much lower acidity than that recommended by earlier workers, and the background colour is greatly reduced by extraction of the colour with *iso*amyl alcohol. *Procedure*—Add a protein hydrolysate (5 ml, containing 0·2 to 0·4 mg of cystine, and $\simeq 1{\cdot}1$ *N* in HCl) to 5 ml of conc. HCl in a boiling-tube; add 0·3 g of zinc dust, cover the tube and shake it until the zinc is dissolved ($\simeq$30 min.); transfer 1 ml of this soln. to a boiling-tube, add 5 ml of reagent [24 g of $ZnSO_4.7H_2O$, 8 g of $(NH_4)_2SO_4.FeSO_4.6H_2O$ and 0·04 g of **I** sulphate in 100 ml of *N* H_2SO_4], and set aside the mixture for 4 min. Add 3 ml of $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$ soln. (20% in *N* H_2SO_4), loosely stopper the tube and immerse to within 5 cm of the top in boiling water for 50 min. Cool, extract with *iso*amyl alcohol (2×10 ml), combine the organic extracts and wash by shaking with aq. NH_3 - NH_4Cl soln. (2×10 ml) (10·7 g of NH_4Cl and 11·2 ml of conc. aq. NH_3 in 100 ml). Filter the alcohol layer through a dry paper and measure its extinction against a blank, with an Ilford 626 filter. The error is $<1\%$. Beer's law is valid up to 40 μg of cystine.

T. R. ANDREW

1866. **Preparation and determination of sodium hydrogen S-(2-aminoethyl) phosphorothioate (sodium hydrogen cysteamine S-phosphate).** S. Åkerfeldt (E. Pennsylvania Psych. Inst., Philadelphia, Pa., U.S.A.). *Acta Chem. Scand.*, 1959, **13** (7), 1479–1480 (in English).—Sodium hydrogen S-(2-aminoethyl) phosphorothioate (**I**) is prepared by reaction of 2-bromoethylammonium bromide with Na_2SPO_3 in aq. dimethylformamide soln. The attachment of the aminoethyl group to the S atom is shown by various methods. The determination of **I** is effected by adding to 0·5 ml of **I** soln. (containing up to 1 μmole) 0·1 ml of a soln. containing 70 mg of mercuric acetate in 100 ml of 5% aq. acetic acid, 2·9 ml of water, 0·50 ml of metol soln. and 1 ml of molybdate soln. (both prepared according to Gomori, *J. Lab. Clin. Med.*, 1942, **27**, 955). After 1 hr. the extinction is determined at 660 mμ. The error is within $\pm 1\%$.

E. J. H. BIRCH

1867. **Biosynthesis of methionine in liver. [Determination of [^{14}C]- or [^{35}S]- methionine in the presence of labelled 1:3-thiazan-4-carboxylic acid (tetrahydro-1:3-thiazine-4-carboxylic acid).]** A. Stevens and W. Sakami (Dept. of Biochem., Western Reserve Univ. Sch. of Med., Cleveland, Ohio, U.S.A.). *J. Biol. Chem.*, 1959, **234** (8), 2063–2072.—The mixture of [^{14}C]- or [^{35}S]-methionine and [2-^{14}C]- or [^{35}S]-tetrahydro-1:3-thiazine-4-carboxylic acid is oxidised with HIO_4, which converts the methionine into its sulphoxide. This is separated from any other radioactive product on a column of Dowex 50. *Procedure*—A trichloroacetic acid supernatant soln. (2 ml) is mixed with 0·3 *M* $NaIO_4$ (0·8 ml) and set aside at room temp. for 15 min. Diacetyl (0·05 ml) is added to destroy the excess of HIO_4; the mixture is shaken and set aside for 10 min. and then the pH is adjusted to 5·0 by the addition of *N* NaOH. A saturated soln. of dimedone (5 ml) is added and after 6 hr. the mixture is filtered and the filtrate is chromatographed on Dowex 50 (H form) with *N* HCl as the eluent. The sulphoxide fraction (90 to 170 ml) is collected and assayed for ^{14}C or ^{35}S by a standard method.

J. N. ASHLEY

1868. **Investigation of a method of quantitative protein determination.** P. Gerhardt Scheurlen (Med. Univ.-Klinik, Tübingen, Germany). *Clin. Chim. Acta*, 1959, **4** (6), 760–766.—The "protein-error" in pH determination with indicators is studied with regard to photometric protein determination in soln. The effect is dependent on the nature of the protein, bovine and human albumin showing much greater protein-error than protein mixtures or pure γ-globulin. It is possible to make accurate protein determinations in the concn. range 30 to 400 μg per ml by photometric measurement of the colour change.

H. F. W. KIRKPATRICK

1869. **Concerning chemistry and testing of Bence-Jones proteins.** B. Jirgensons, T. Ikenaka and V. Gorguraki (M. D. Anderson Hosp. and Tumor Inst., Houston, Tex., U.S.A.). *Clin. Chim. Acta*, 1959, **4** (6), 876-882.—The amino-acid composition and terminal amino groups of two typical individual specimens are determined. The proteins showed a characteristic propanol flocculation and the following test is proposed. Urine (1 to 4 ml according to the concn. of protein) is mixed with various amounts of propanol. The final vol. in each tube, the amount of protein, and the pH are constant in a single series. The pH of the urine is adjusted to 4·8 to 5·0 by adding acetic acid. The presence of Bence-Jones protein is indicated by a turbidity peak at 15 to 25% v/v of propanol in a plot of turbidity against propanol concn. at 20° to 25°. Temp. effects are pronounced.

H. F. W. KIRKPATRICK

1870. **Amperometric titration of disulphide and sulphydryl [mercapto groups] in proteins in 8 *M* urea.** J. R. Carter (Dept. of Path., Coll. of Med.,

State Univ. of Iowa, U.S.A.). *J. Biol. Chem.*, 1959, **234** (7), 1705–1710.—The protein is dissolved in 8 *M* urea which "unfolds" the molecule and the disulphide bonds are then caused to react with Na_2SO_3 which releases one mole of mercapto compound for each mole of disulphide. The mixture is then titrated with standard $AgNO_3$ soln., with a platinum indicator electrode and a mercury - mercuric oxide - satd. barium hydroxide reference electrode to give the sum of mercapto groups originally present and those formed by the opening of the disulphide linkages. For the determination of the original mercapto groups only, Na_2CO_3 is used instead of Na_2SO_3 and the titration is carried out with $AgNO_3$ as before. J. N. ASHLEY

1871. **Albumin diagnostic composition.** Miles Laboratories, Inc. Brit. Pat. 826,066; date appl. 13.5.57. U.S.A., date appl. 21.5.56.—Strips of filter-paper are impregnated with an indicator dye which exhibits "protein-error" (*e.g.*, bromophenol blue or tetrabromophenolphthalein ethyl ester), and an acid-reacting buffer (*e.g.*, Na citrate - citric acid) that buffers the dye at a pH value close to, but on the acid side of, the pH at which the dye normally changes colour. J. M. JACOBS

1872. **Thiobarbituric acid assay of sialic acids** L. Warren (Nat. Inst. of Arthritis and Metabol. Diseases, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1959, **234** (8), 1971–1975.—A sensitive and specific assay is described, which determines only free sialic acids. These are oxidised with $NaIO_4$ in conc. H_2SO_4, and the oxidation product (probably β-formylpyruvic acid) is condensed with thiobarbituric acid; the resulting chromophore is extracted with *cyclo*hexanone, and then determined spectrophotometrically at 549 mμ (for N-acetylneuraminic acid ϵ is 57,000). For the determination of bound sialic acids, the tissues or homogenates are hydrolysed for 1 hr. with 0·1 *N* H_2SO_4 at 80°, and the assay is carried out on the hydrolysate. Other methods for the determination of sialic acids are one-twelfth as sensitive. J. N. ASHLEY

1873. **Quantitative determination of neuraminic acids in cerebrospinal fluid.** G. Chistoni and R. Zappoli (Clin. della Malattie Nervose e Mentali, Univ. di Modena, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (12), 707–717.—Cerebrospinal fluid (**I**) contains neuraminic acid derivatives (**II**) bound to glyco- and muco-proteins. Methods for the determination of **II**, depending on the orcinol reaction, suffer from interference by substances such as hexoses. Thus, the colour produced by N-acetylneuraminic acid (**III**) has max. absorption at 580 mμ, at which point interference from 0·025% of glucose or fructose is appreciable. In the following method, interfering substances were eliminated by passage through ion-exchange columns. **I** (4 ml) was hydrolysed with 0·05 *N* H_2SO_4 (5 ml) for 1 hr. at 85°. The cold hydrolysate was passed successively through columns of Dowex 50 and Dowex 2, which were then washed with water (10 ml). The **II** were eluted successively from the Dowex-2 column with acetate buffer at pH 4·6 (8 ml), the eluate was diluted to 10 ml, and aliquots (4 ml) were heated at 100° for 15 min. with Bial's orcinol reagent (2 ml). After cooling, the coloured product was extracted into amyl alcohol (5 ml) and the extinction of the extract was measured at 580 mμ, against a blank in which water (4 ml) was submitted to the procedure described above. Total **II** was then obtained by reference to a curve previously constructed by submitting **III** (1 to 7 mg per 100 ml) to the Bial procedure. Free **II** was determined on **I** submitted to the procedure described above, but with the hydrolysis omitted. The total **II** in the **I** of 15 normal subjects was determined as 0·9 to 2·7 mg per 100 ml, the free **II** fraction being 7·5 to 9·4% of the total in 6 cases. C. RAINBOW

1874. **Paper-chromatographic determination of histidine or urocanic acid.** T. A. Goryukhina. *Ukr. Biokhim. Zhur.*, 1959, **31** (1), 138–143; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (19), Abstr. No. 25,013.—The solvent used is *n*-butanol - glacial acetic acid - H_2O (4:1:5), and the locating agent comprises a mixture of (*a*) sulphanilic acid (1 g), HCl (sp. gr. 1·19) (10 ml) and H_2O to 100 ml (3 ml) and (*b*) freshly prepared 5% $NaNO_2$ soln. (3 ml), to which are added after 5 min. a further 12 ml of soln. (*a*) and H_2O to 100 ml. The wet chromatogram is exposed to fumes of aq. NH_3 soln. (25%). The spot due to urocanic acid (R_F 0·7 to 0·73) is yellow - orange, and that due to histidine (R_F 0·21) is bright red. For a quantitative determination, the corresponding zones from a second chromatogram are cut out and eluted with H_2O (2 ml). The diazo reagent (1·2 ml), as used for locating, is added, followed after 10 min. by 10% Na_2CO_3 soln. (0·6 ml); 30 min. later the soln. is measured photometrically with a green filter. The method is sensitive to 3 μg of histidine or urocanic acid. K. R. C.

1875. **Determination of L-5-vinyl-2-thio-oxazolidone [L-5-vinyl-2-oxazolidinethione] from plant material and milk.** M. Kreula and M. Kiesvaara (Lab. of Valio, Biochem. Inst., Helsinki, Finland). *Acta Chem. Scand.*, 1959, **13** (7), 1375–1382 (in English).—The determination of small amounts of L-5-vinyl-2-oxazolidinethione (**I**) in green plants, silage, seeds and milk is described. *Procedure*—Minced plant material (10 g) is kept at room temp. for 7 hr., heated for 5 min. on a water bath and pressed. The filtered expressed liquid (1·5 ml) is treated with 0·5 ml of phosphate buffer (pH 6·5) and extracted with ethyl acetate satd. with water (2 × 5 ml). The combined extracts are evaporated and the residue is dissolved in 0·5 ml of hot water. Silage is prepared similarly, but, as it is acid, it is adjusted to pH ≃6 before adding the buffer. Seeds are finely ground, moistened and treated similarly. Two-dimensional paper chromatograms are run on 0·25 ml of the final aq. soln. with methanol - water and then methanol - ether (1:1). The spot of **I** (identified under u.v. light) is cut out and extracted with hot water, and the extinctions are measured at 220, 240 and 260 mμ, correcting for impurity by subtracting the mean of the extinctions at 220 mμ and 260 mμ from that at 240 mμ and multiplying by 1·16 (*cf.* Astwood *et al.*, *J. Biol. Chem.*, 1949, **181**, 121). The amount of **I** in milk is determined by heating 300 ml of milk to 85°, cooling rapidly, and extracting with two 500-ml portions of ethyl acetate. The extracts are evaporated almost to dryness and treated 4 times with 5 ml of 1% aq. NH_3, with warming. The combined ammonia extracts are adjusted to pH 6·5 to 7·5 and milk fat is removed with 5 ml of light petroleum. The aq. phase is extracted with $CHCl_3$ and evaporated to dryness; the residue is dissolved in 0·5 ml of water and 0·3 ml of this soln. is then subjected to two-dimensional chromatography with 0·5% acetic acid followed by methanol - ether (1:1). The extinction is determined between 220 and 290 mμ and corrected by multiplying the height of the max.

at 240 mμ above the straight line joining the extinctions at 225 and 260 mμ by 1·16. A similar method is described for skim milk, the solvents being 0·5% acetic acid and methanol - water. Extinctions are converted to μg per ml by multiplying by 7·5. The results are discussed with respect to the enzymatic formation and the recovery of **I** in the materials.
E. J. H. BIRCH

1876. **High-resolution infra-red spectra of steroids in the carbon-hydrogen stretching region.** A. M. Smith and C. R. Eddy (Eastern Reg. Res. Lab., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1539–1542.—The i.r. spectra of 40 steroidal sapogenins were investigated in the 3100 to 2750-cm^{-1} region, with a double monochromator and lithium fluoride prisms, in order to correlate absorption in the C–H stretching region with structural features of the molecule. Of the seven to nine bands observed, five can be associated with specific modes of CH, CH_2 and CH_3 groups. Most of the bands are very sensitive to structural changes in the carbon skeleton. It is frequently possible to distinguish between 25D and 25L configurations, detect openings of E and F rings and determine the configuration of the CH_3 group in the 20 position.
G. P. COOK

1877. **Separation and chemical assay of lipid classes.** E. Van Handel (Physiol. Div., Univ. of Tennessee, Memphis, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1959, **36** (7), 294–297.—Micro-determinations of cholesterol, triglycerides and phospholipids are discussed, and a method for determining total cholesterol in serum is given. *Procedure*—A mixture of 3% of toluene-*p*-sulphonic acid and 25·5% of acetic acid in acetic anhydride (4 ml) is added to 0·2 ml of plasma and stirred. A reference standard containing 0·4 mg of cholesterol and 0·2 ml of water is similarly treated. After cooling the soln., 0·4 ml of H_2SO_4 is rapidly added and mixed, and the soln. is cooled to room temp. The extinction is measured at 625 mμ or 550 mμ after 40 to 60 min. A serum blank is obtained by adding 4·4 ml of a 12% soln. of toluene-*p*-sulphonic acid in acetic acid to 0·2 ml of serum. Cholesterol can be separated from its esters and triglycerides by passing the mixture (> 20 mg) dissolved in light petroleum (boiling-range 60° to 70°) through a column (40 cm × 1 cm) containing a mixture of 0·5 g of Super-Cel and 0·5 g of a non-activated silicic acid, and eluting with 20 ml of $CHCl_3$ - light petroleum (1:9). The eluate contains the cholesteryl esters; triglycerides and free cholesterol are then removed by eluting with 20 ml of $CHCl_3$.
G. R. WHALLEY

1878. **Spectrophotometric determination of cholic acid in deoxycholic acid.** R. Willemart and J. Ferdet (Serv. Sci., Roussel-Uclaf, France). *Ann. Pharm. Franç.*, 1959, **17** (5), 366–367.—Dehydration of cholic acid with H_3PO_4 affords a compound that absorbs at 260 mμ, and is not influenced by deoxycholic acid. *Procedure*—The sample of deoxycholic acid (< 0·1 g) is dissolved in 2 ml of 0·2 *N* aq. NaOH, and 10 ml of H_3PO_4 (*d* 1·71) is added. After heating the soln. for 90 min. at 40°, cooling and making up to 25 ml with butanol, the extinction is read after 5 min. at 260 mμ against a standard.
E. J. H. BIRCH

1879. **Separation and quantitative estimation of pregnane-3α:17α:20α-triol, 3α:17α:20α-trihydroxy-5β-pregnan-11-one and other urinary acetaldehydogenic steroids.** R. I. Cox (Dept. of Vet. Physiol., Univ. of Sydney, Australia). *J. Biol. Chem.*, 1959, **234** (7), 1693–1697.—The separation and determination of 5β-pregnane-3α:17α:20α-triol, 5β-pregnane-3α:17α:20β-triol, pregn-5-ene-3β:17α:-20α-triol, 5α-pregnane-3α:17α:20α-triol, 3α:17α:-20α-trihydroxy-5β-pregnan-11-one, and 5β-pregnane-3α:11β:17α:20α-tetrol on paper chromatograms are described. The paper is impregnated with formamide - methanol (1:1) and, after application of the urine or soln. of the steroids, the solvent system used is either benzene - formamide or dichloroethane - formamide; 70% H_3PO_4 is used to detect the steroids, and after being dried and heated at 87° to 90° for 10 min. each spot has a characteristic colour in daylight and a fluorescence in u.v. light. The determinations in extracts of urine or on sections of paper are based on the fact that all these steroids contain a propylene glycol type of side-chain and when oxidised with HIO_4 they give rise to acetaldehyde which is aerated into 1% aq. $NaHSO_3$ soln. (0·5 ml). The soln. is then treated with conc. H_2SO_4 (3·5 ml), 4% aq. $CuSO_4$ soln. (0·02 ml), and 1·5% 4-hydroxydiphenyl soln. in 0·1 *N* NaOH (0·02 ml), a temp. of 0° being maintained throughout the additions. After 1 hr. at 0°, the tubes are heated at 100° for 1·5 min. to destroy the excess of 4-hydroxydiphenyl and the extinction is determined spectrophotometrically at 565 mμ and the amounts are obtained from standard graphs.
J. N. ASHLEY

1880. **Determination of lactic dehydrogenase.** M. Ševela and J. Továrek (Inst. Med. Chem., Med. Fac., Univ., Brno, Czechoslovakia). *Čas. Lék. Čes.*, 1959, **98** (27), 844–848.—The enzyme activity is calculated from the amount of pyruvate formed during 15 min. at 37° in a medium containing 4·45 *M* Na lactate (0·2 ml), 0·003 *M* diphosphopyridine nucleotide (0·3 ml) and 0·1 ml of the diluted sample (1:5). The pH of the medium is maintained at 8·8 by a 0·045 *M* buffer soln. of tri(hydroxymethyl)methylamine or a 0·025 *M* soln. of $Na_4P_2O_7$. The pyruvate is determined photometrically with 2:4-dinitrophenylhydrazine in an alkaline medium.
J. ZÝKA

1881. **DDT-dehydrochlorinase. I. Isolation, chemical properties, and spectrophotometric assay.** H. Lipke and C. W. Kearns (Dept. of Entomol., Univ. of Illinois, Urbana, U.S.A.). *J. Biol. Chem.*, 1959, **234** (8), 2123–2128.—The enzyme is incubated with DDT, which has been solubilised by egg-yolk lipoprotein, and reduced glutathione at 25°. The extinction of the ethylene derivative formed by dehydrochlorination is measured spectrophotometrically at 260 mμ.
J. N. ASHLEY

1882. **Comparative biochemistry of urea synthesis. I. Methods for the quantitative assay of urea-cycle enzymes in liver.** G. W. Brown, jun., and P. P. Cohen (Dept. of Physiol. Chem., Univ. of Wisconsin Sch. of Med., Madison, U.S.A.). *J. Biol. Chem.*, 1959, **234** (7), 1769–1774.—Complete assays for the five enzymes of the urea cycle are described. They can by carried out on frog-liver extracts equiv. to ≃150 mg of wet liver. Cell-free extracts are prepared with the aid of 0·1% cetyltrimethylammonium bromide soln. which gives extracts of higher specific activity than does water alone. The first extract contains arginine synthetase and the argininosuccinate cleavage enzyme; assay systems are given for each enzyme, and in both cases the urea formed is determined by a modification of Archibald's method (*Brit. Abstr. C*, 1945, 135), which involves

the condensation of the urea with α-oximinopropiophenone to give a product with an absorption max. at 540 mμ. The first and second extracts contain carbamyl phosphate synthetase, ornithine transcarbamylase, and arginase, and these are determined in a mixture of equal volumes of the two extracts. By use of the appropriate assay systems, the arginase is determined by the amount of urea formed, whilst the citrulline formed by the other two enzymes is determined by the same modification of Archibald's method, which involves the condensation of citrulline with diacetyl monoxime to give a product with max. absorption at 490 mμ. J. N. ASHLEY

1883. **Bacterial oxidation of steroids. II. Studies on the enzymic mechanism of ring-A dehydrogenation. [Determination of Δ-dehydrogenase activity.]** H. R. Levy and P. Talalay (Ben May Lab. for Cancer Res., Univ. of Chicago, Ill., U.S.A.). *J. Biol. Chem.*, 1959, **234** (8), 2014–2021.—Three separate methods are described. The first involves the measurement of differences in u.v. absorption. The conversion of androstane-3:17-dione into androsta-1:4-diene-3:17-dione by enzyme preparations from *Pseudomonas testosteroni* is associated with a marked increase in u.v. absorption. This method is not suitable for the determination of Δ^1- and Δ^4-5α-dehydrogenase activities singly, but will give an over-all measure of the two activities. The reaction is stopped by adding aq. HCl and the steroids are extracted with dichloromethane. The extract is dried over anhyd. Na_2SO_4, and the extinction is measured spectrophotometrically against a control at 240 mμ. The second method involves the measurement of phenol formation. The conversion of 19-nortestosterone or oestr-4-ene-3:17-dione into oestrone is determined by measuring the formation of the phenolic steroid with Folin's reagent. This is a very sensitive and specific assay for the Δ^1-dehydrogenase, and as little as 0·02 μmole of the phenol can be determined accurately. The use of oestr-1-ene-3:17-dione as a substrate for the assay of Δ^4-5α-dehydrogenase activity is less practical because this steroid is oxidised only slowly. The third method is based on the direct measurement of dye reduction. The anaerobic reduction of phenazine methosulphate during the oxidation of androst-4-ene-3:17-dione by Δ^1-5α-dehydrogenase or of androst-1-ene-3:17-dione by Δ^4-5α-dehydrogenase is followed by measurement of the extinction at 430 mμ. Under defined conditions the extent of reduction is proportional to the activity of the enzyme. This is the simplest and most convenient method. J. N. ASHLEY

See also Abstracts—**1705,** Determination of ammonium salts. **1711,** Orthophosphate in biological products. **1787,** Electrophoresis of carbo-hydrates.

Pharmaceutical analysis

1884. **Testing the purity of pharmaceutical raw materials with the aid of micro-methods.** H. Lehner (Res. Inst., Dr. A. Wander A.-G., Bern, Switzerland). *Chimia*, 1959, **13** (8), 248–252.—Purity tests are discussed from the pharmacopoeial and industrial points of view. The use of quant. (including polarographic and spectrophotometric) methods is mentioned and illustrated by reference to the determination of traces of xanthone (<0·02%) in 9:2′-piperidinoethylxanthen methobromide (polarographically at pH 6·88), of toxic chlorinated aromatic by-products (5×10^{-5} *M* Cl, by combustion in O and potentiometric titration in 50% *iso*propyl alcohol, 0·05 *N* with respect to HNO_3), and of Mg (20 μg per g in salts of sodium or calcium by adsorption on a cation-exchange resin, *e.g.*, Dowex 50-W, and elution with *N* HCl, which separates the Mg as a middle fraction). J. P. STERN

1885. **Use of radioactive isotopes in pharmaceutics.** K. E. Schulte (Isotopen-Lab., Abt. II d. Pharm. Inst., Freien Univ., Berlin) *Arzneimittel-Forsch.*, 1959, **9** (6), 379–388.—A review. Topics briefly discussed include the use of radioactive isotopes in the determination of solubilities and the isotope dilution method of analysis as applied to pharmaceutical preparations. (39 references.) A. G. COOPER

1886. **Conditions for the precipitation of alkaloids and synthetic bases with Reinecke's salt. I. Alkaloids.** W. Poethke, P. Gebert and E. Müller (Inst. für Pharm. u. Lebensmittelchem., Friedrich-Schiller-Univ., Jena). *Pharm. Zentralh.*, 1959, **98** (7), 389–400.—Optimum pH conditions are given for the quant. pptn. of mono- and di-reineckates of quinine, quinidine, cinchonine, cinchonidine and sparteine. A. R. ROGERS

1887. **Rapid isolation and identification of toxic nitrogenous bases. I. Use of tungstosilicic acid in the isolation of alkaloids and analogous toxic compounds.** Ming-Chue Huang, Nai-Chao Hu, Chen-Weh Loo, Ti-Tzen Wang, Tzun-Fu Wu, Lan-Chang Ling and Yin-Hwa Shong (Dept. of Pharm., Secondary Military Med. Coll., Shanghai). *Acta Pharm. Sinica*, 1959, **7** (8), 287–294.—Tungstosilicic acid (**I**) has been used as a pptg. agent in the isolation of alkaloids and toxic nitrogenous bases from biological materials. Conditions of pptn. such as pH, concn. of HCl and amount of **I** are examined. A pH of 1·2 to 1·8 is recommended. The method described is more rapid and simpler than the Stas - Otto method. *Procedure*—Mix urine or a dil. HCl extract of finely minced meat (5 ml) (≃5 to 10 mg of alkaloid) in a beaker with 10% HCl (4 to 8 drops) and 5% of **I** (2·5 to 5 ml), and immerse the beaker in a boiling-water bath for 5 min. Cool and separate the ppt. by centrifuging. Dissolve the ppt. in conc. aq. NH_3 (8 drops) and extract the alkaloid 3 times with a suitable solvent. Dry the combined extracts with anhyd. Na_2SO_4, filter and distil off the solvent. The residue is ready for identification. Experiments show that 70% to 92% of morphine is recovered by the proposed procedure from dil. HCl soln. and 60% to 75% from urine. Recoveries of procaine from dil. HCl soln. or from urine are 80% to 100%. Concn. limits and minimum amounts of 23 alkaloids and toxic nitrogenous bases pptd. by **I** are given. S. H. YUEN

1888. **Photometric determination of some organic nitrogenous bases.** J. Čelechovský and D. Svobodová (Phys.-Anal. Dept., Pharm. Fac., Univ. Brno, Czechoslovakia). *Českosl. Farm.*, 1959, **8** (7), 380–384.—Certain organic bases, mainly alkaloids, can be pptd. with a soln. of molybdophosphoric acid; the excess of the reagent is masked with tartrate and the content of Mo^{VI} in the ppt. is determined photometrically after dissolution and reduction to molybdenum blue. The concn. conditions for reproducibility depend on the strength of the base and on the solubility of the ppt. *Procedure for phenazone*—To the soln. of the reagent

($6{\cdot}33 \times 10^{-3}$ *M*) (5 ml) add phenazone soln. (1 to 5 mg in 5 ml) and then *M* tartaric acid (5 ml). After 8 min. reduce with a freshly prepared soln. of $SnCl_2$ (to 0·15 g of tin add 3 ml of conc. HCl and a few drops of 5% $CuSO_4.5H_2O$ soln. as a catalyst; dissolve on a water bath, filter and dilute to 100 ml with H_2O) (10 ml) and dilute to 50 ml. Mix till dissolution is complete, then measure the extinction at 750 mμ and compare with a calibration curve. Pilocarpine, caffeine and morphine can be determined similarly. The determination of amidopyrine is vitiated by its reducing properties.

J. ZÝKA

1889. **Chromatographic detection of opium in tea and other foods.** P. N. Sengupta, S. N. Mitra and B. R. Roy (Central Food Lab., Calcutta). *J. Instn Chem., India*, 1959, **31** (3), 124–125.—*Procedure*—The test soln. is spotted on to a circular Whatman No. 1 filter-paper and is developed with the butanol phase of a soln. prepared by shaking *n*-butanol or *iso*butyl alcohol several times during 3 days with ethanol - glacial acetic acid - water (10:1:5) and allowing to separate. After development the paper is dried and sprayed with a soln. of $PtCl_4$ (1 g) in water (10 ml) mixed with 4% KI soln. (250 ml) and made up to 500 ml. Opium gives immediate bluish-green streaks on a reddish-brown background.

C. A. SLATER

1890. **Ion-exchange separation method for microdetermination of tropane alkaloids in the presence of morphine.** E. Sjöström and A. Randell (Anal. Lab., Lääketehdas Orion Oy, Helsinki, Finland). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (8), 445–448.—Transfer a methanolic soln. of the sample to a column of Dowex-1 resin (OH^- form) and collect the percolate. If the sample contains glycerol, pass the percolate through a column of Dowex 50X-4 resin (H^+ form) and elute the tropane alkaloids from the second column with methanolic NH_3. Determine tropane alkaloids in an aliquot of the eluate by the diazotisation procedure of Nir-Grosfield and Weissenberg (*cf. Anal. Abstr.*, 1958, **5**, 3106), with slight modifications. The error is about ±5%.

A. R. ROGERS

1891. **Spectrophotofluorimetry of reserpine, other rauwolfia alkaloids and related compounds.** R. P. Haycock, P. B. Sheth and W. J. Mader (Res. Dept., Ciba Pharmaceutical Products Inc., Summit, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (8), 479–485.—The fluorescent light emitted at 360 mμ by a methanolic soln. of reserpine excited by incident light of wavelength 280 mμ is directly proportional to concn. in the range 1 to 5 μg per ml. This forms the basis of a method for the determination of reserpine in admixture with rescinnamine, which shows different excitation and fluorescence spectra and causes no interference. The error is $< \pm 3\%$.

A. R. ROGERS

1892. **New method of evaluating the microbiological test for cobalamins.** J. Černá (Res. Inst. Food Technol., Prague). *Českosl. Farm.*, 1959, **8** (7), 376–379.—The method is based on the quant. evaluation of plate-assays by the method of standard addition. The method can also be used for the analysis of samples that cause the inhibition of the growth of the micro-organism. A simple mathematical expression is given for the calculation of the cobalamin concn. of the sample. The coeff. of variation (95% confidence level, 20 analyses) is ±24%.

J. ZÝKA

1893. **Gravimetric determination of insulin. The study of initiating gels.** F. Horák, J. Gašperík, J. Šafář and H. Pelzová (Dept. Org. Technol., Tech. Univ., Bratislava, Czechoslovakia). *Českosl. Farm.*, 1959, **8** (5), 208–211.—*Procedure*—Dissolve the sample (0·5 g) in H_2O (acidified with few drops of *N* HCl) and dilute to 250 ml. Adjust to pH 2 and filter (G3 filter washed with 20% aq. NH_3). Divide the soln. into 80-ml centrifuge tubes. To the soln. of the sample (60 ml) add H_2O (15·7 ml), H_2SO_4 (20%) (1 ml) and a dilute soln. of fibrils (2 ml of insulin gel diluted with 20 ml of 0·05 *N* HCl) (3·3 ml) and heat at 48° for 20 hr. Add another vol. (3·3 ml) of dilute soln. of fibrils, repeat the heating for 20 hr. and centrifuge (45 min.). Remove the supernatant liquid, transfer the pptd. fibrils into a 10-ml test-tube and wash (*i*) once with 5 ml of a mixture of ethanol (63%) (99 ml), *N* HCl (1 ml) and NH_4Cl (1 g); (*ii*) once with 5 ml of a mixture of ethanol (95%) (99 ml) and *N* HCl (1 ml); (*iii*) once with 5 ml of a mixture of H_2O (99 ml) and *N* HCl (1 ml); and (*iv*) once with 5 ml of (*ii*) soln. Wash the residue with purified acetone (3 × 5 ml), remove the solvent, dry the residue over P_2O_5 and weigh.

J. ZÝKA

1894. **Rapid titration of antibiotics by means of resazurin.** G. De Felip, S. Alberti and L. von Lorch (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1959, **22** (5), 534–542.—This cylinder - plate method is based on the use of resazurin incorporated in an agar medium as an indicator of bacterial growth. Satisfactory readings of the diameters of the zones of inhibition are obtained in 3 to 6 hr.

L. ZANONI

1895. **Contribution to the analysis of oxytetracycline.** E. Hannig and H. Heyroth-Straube-Kögler (Pharm. Inst. d. Martin-Luther-Univ., Halle-Wittenberg). *Pharm. Zentralh.*, 1959, **98** (7), 385–389.—Tetracycline, chlortetracycline and oxytetracycline (**I**) may be identified and distinguished by their colour reactions with a soln. of *p*-dimethylaminobenzaldehyde in H_2SO_4, alone and after dilution with H_2O. The reaction may also be used for the quant. determination of **I**. *Procedure*—Evaporate to dryness a soln. of **I** hydrochloride (200 to 800 μg) in methanol and dissolve the residue in a freshly prepared soln. of *p*-dimethylaminobenzaldehyde in conc. H_2SO_4 - H_2O (19:1) (4 ml). Heat on a boiling-water bath for 15 min., cool, dilute to 25 ml with conc. H_2SO_4 and measure the extinction in a 0·5-cm cell in a Pulfrich photometer with filter S57.

A. R. ROGERS

1896. **Psicofuranine. I. Discovery, isolation and properties.** T. E. Eble, H. Hoeksema, G. A. Boyack and G. M. Savage (Upjohn Co., Kalamazoo, Mich., U.S.A.). *Antibiot. & Chemother.*, 1959, **9** (7), 419–420.—The chemical and physical properties, including the i.r. spectrum and optical rotation, of a new antibiotic, psicofuranine (6-amino-9-D-psicofuranosylpurine) are given. In 0·01 *N* acid, $E^{1\%}_{1\,cm}$ = 508 at 259 mμ and, in 0·01 *N* alkali, 527 at 261 mμ.

IV. Microbiological assay. L. J. Hanka, M. R. Burch and W. T. Sokolski. *Ibid.*, 1959, **9** (7), 432–435.—A paper disc - agar diffusion method is described, with *Staphylococcus aureus* F.D.A.-209P as test organism. The method is suitable for concn. of 10 to 80 μg per ml in water, each 12·7-mm disc carrying 0·08 ml of soln. An adaptation of the method is suitable for the detection of 3 μg per ml

in blood or serum. With the turbidimetric modification described, the sensitivity is 0·4 to 2·0 μg per ml.

V. Paper chromatography and ultra-violet absorption assay. W. T. Sokolski, N. J. Eilers and T. E. Eble. *Ibid.*, 1959, **9** (7), 436–438.—The behaviour of psicofuranine and related compounds on paper developed with six different solvent systems is studied and a paper-strip assay method is described for 1 to 20 μg of material, determined by scanning at 262 mμ in a recording spectrophotometer. The standard error of an assay in triplicate is 16%. W. H. C. SHAW

1897. **Determination of some pharmaceutical products in non-aqueous medium.** P. Kerny, J.-P. Billon and F. Bigeard (Soc. des Usines Chim., Rhône-Poulenc, Lab. de Rech. Anal., Vitry-sur-Seine, France). *Ann. Pharm. Franç.*, 1959, **17** (4), 284–294.—Operating conditions, indicators and preparation of reagents are described for the titration of acetylcholine chloride, adrenaline, amidopyrine, amylocaine hydrochloride, chlorpromazine hydrochloride, choline chloride, ephedrine, ephedrine hydrochloride, pethidine hydrochloride, promethazine hydrochloride, sodium gentisate, sodium 4-aminosalicylate, amethocaine hydrochloride, thiamine and pyridoxine as bases with $HClO_4$ in acetic acid or acetone. Operating conditions, indicators and preparation of reagents are described for the titration of barbitone, butobarbitone, mebubarbital, methylphenobarbitone, phenobarbitone, sulphacetamide, sulphadiazine, sulphamerazine, sulphapyridine, sulphathiazole and theophylline as acids in dimethylformamide with potassium methoxide (standardised with benzoic acid). A simple apparatus is described for the potassium methoxide titration in the absence of CO_2. E. J. H. BIRCH

1898. **Determination of acetylsalicylic acid, phenacetin and caffeine in tablets.** C.-M. P. Wirth (Sydney Ross Co. S.A., Mexico City). *Pharm. Acta Helv.*, 1959, **34** (7), 283–295 (in German).—To determine acetylsalicylic acid, suspend the powdered sample (0·5 g) in neutral 95% ethanol and titrate with 0·25 *N* NaOH, with phenolphthalein as indicator. Apply a correction for the quantity of free salicylic acid present, as determined by the method of the U.S.P. XV. To determine phenacetin, shake the powdered sample (containing ≃20 mg of phenacetin) with 95% ethanol (10 ml) and filter; heat 3 ml of the filtrate with conc. HCl (3 ml), evaporate to a vol. of ≃1 ml, cool and dilute with H_2O to 50 ml; set aside a 2-ml aliquot with 50% ammonium citrate soln. (8 ml) and 1% chromic acid soln. (0·1 ml) for 7 min. at 24° and measure the extinction at 543 mμ against a reagent blank. To determine caffeine, shake the powdered sample (4 g) vigorously with H_2O (100 ml) and filter; mix 50 ml of the filtrate with 10% H_2SO_4 soln. (5 ml) and 0·1 *N* iodine (25 ml), dilute with H_2O to 100 ml, mix and filter; titrate 50 ml of the filtrate with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. The complete analysis takes 1·5 hr. A. R. ROGERS

1899. **A rapid and precise method of determining some amino-alcohol esters.** F. Gélébart (Lab. Dausse, Usine de Morigny-Champigny, Seine-et-Oise, France). *Ann. Pharm. Franç.*, 1959, **17** (4), 278–284.—Certain anaesthetics and anti-spasmodics which are esters of diethylaminoethanol can be determined by distillation from standard NaOH, when the released base distils in the water vapour and is received in standard acid. *Procedure for procaine hydrochloride*—The sample (0·20 to 0·25 g) is introduced into a 250-ml flask with 75 ml of H_2O and 25 ml of 0·1 *N* NaOH, and 75 ml is distilled over in 25 min. and received in 20 ml of 0·1 *N* H_2SO_4. The excess of acid in the distillate is determined with 0·1 *N* NaOH, with methyl red as indicator. To the contents of the distillation flask is added 25 ml of 0·1 *N* H_2SO_4, and the excess of acid is determined with 0·1 *N* NaOH (phenolphthalein as indicator). The NaOH titre is equivalent to the *p*-aminobenzoic acid produced in the hydrolysis. The two figures are in agreement for the procaine content. A 10-fold concn. of reactants and sample is acceptable. For other diethylaminoethanol esters the higher concn. leads to pptn. of the unhydrolysed base and it is necessary to complete the hydrolysis by heating under reflux before distillation. Other amino-alcohols (including dimethylaminoethanol and piperidinoethanol) are not fully entrained in the water vapour, and these esters cannot thus be determined. Alkaloids present with these substances can be extracted and determined separately. E. J. H. BIRCH

1900. **Determination of some derivatives of glutarimide, glutethimide, bemegride and Aturban (phenglutarimide).** E. Szabolcs and G. Vastagh (Chem. Abteil. d. Staatl. Inst. f. Hygiene, Budapest). *Pharm. Zentralh.*, 1959, **98** (7), 410–417.—Prepare a soln. of glutethimide (60 to 80 mg) in ethanol (10 ml), or a soln. of phenglutarimide hydrochloride (50 to 80 mg) in H_2O (10 ml), boil under reflux for 5 min. with 0·1 *N* NaOH (10 ml), cool, and titrate with 0·1 *N* HCl, with methyl red as indicator. To determine bemegride, dissolve the sample (75 to 100 mg) in 5% NaOH soln. (1 ml), dilute with H_2O (20 ml) and titrate with 0·1 *N* $AgNO_3$. To determine phenglutarimide base, dissolve the sample (50 to 80 mg) in warm ethanol (10 ml), cool, add H_2O (10 ml) and 10% NaOH soln. (0·5 ml) and titrate with 0·1 *N* $AgNO_3$. The results are in general accurate to within ±0·4%. A. R. ROGERS

1901. **Identification and differentiation of sympathomimetic amines.** L. G. Chatten and L. Levi (Dept. of Nat. Health and Welfare, Ottawa, Ontario, Canada). *Anal. Chem.*, 1959, **31** (9), 1581–1585.—The reactions of some sympathomimetic amines with benzoyl chloride, *p*-nitrobenzoyl chloride, benzenesulphonyl chloride, picric acid and ammonium reineckate were studied. By means of colour and micro-crystal tests and the formation of derivatives, it is possible to differentiate dexamphetamine, amphetamine, methamphetamine and ephedrine. Photomicrographs and i.r. absorption spectra of the reaction products can also assist the differentiation and identification of these drugs. G. P. COOK

1902. **Analytical control of meprobamate by non-aqueous titration.** B. Salvesen and O. Solli (Inst. of Pharm., Univ. Oslo, Norway). *Medd. Norsk Farm. Selsk.*, 1959, **21** (7), 85–87 (in English).—Meprobamate is determined by titrating the NH_4Cl formed by acid hydrolysis with $HClO_4$ in glacial acetic acid soln. Gently boil the sample (60 to 110 mg) under reflux with 35% aq. HCl (40 ml) for 45 min. and evaporate almost to dryness. Add glacial acetic acid (10 ml), titrate with a 5% soln. of mercuric acetate in glacial acetic acid to the violet colour of crystal violet, add dioxan (20 ml) and then titrate with 0·1 *N* $HClO_4$ in glacial acetic acid to a bright blue end-point. The NH_4Cl content

of the hydrochloric acid is determined by means of a blank, and the mercuric acetate is tested for its uptake of $HClO_4$. A. R. ROGERS

1903. **The analytical behaviour of some derivatives of phenylacetic acid, imidazoline and benzhydryl ether. I. Colour and precipitation reactions.** M. Hädicke (Staatl. Inst. f. Arzneimittelprüfung, Jena). *Pharm. Zentralh.*, 1959, **98** (7), 377–385.—Reactions with 25 reagents are tabulated for *iso*pentyl phenylaminoacetate hydrochloride, diphenhydramine, hydrochloride, and methobromide, methimazole, tolazoline hydrochloride and naphazoline hydrochloride. A. R. ROGERS

1904. **Assay of succinylcholine chloride in aqueous solution.** R. Okken and F. H. de Haas (Poviet Producten N.V., Amsterdam, Netherlands). *Pharm. Weekbl.*, 1959, **94** (15), 489–496.—Parenteral soln. of suxamethonium chloride (**I**) may contain succinylmonocholine chloride (**II**), succinic acid and choline chloride due to hydrolysis during sterilisation or storage. In the U.S.P. assay method the total esters (calculated as **I**) are determined by hydrolysis. By combining the U.S.P. method with the colorimetric method of Hestrin (*J. Biol. Chem.*, 1949, **120**, 249) it is possible to determine **II** as well as **I** because of the higher extinction of **II** as compared with **I**. M. J. MAURICE

1905. **Volumetric determination of 3-(2-tolyloxy)-propane-1:2-diol [mephenesin] and 3-(2-methoxyphenoxy)propane-1:2-diol [Resyl] with periodic acid.** T. Waaler and R. B. Paulssen (Dept. of Galen. Pharm., Univ. Pharm. Inst., Oslo, Norway). *Medd. Norsk Farm. Selsk.*, 1959, **21** (8), 97–104.—These drugs can be accurately determined by treatment in 0·05 *M* soln. with an excess of 0·05 *M* HIO_4 for 10 min., and back-titration of the residual HIO_4 with arsenite. The sole oxidation products are formaldehyde and the respective aryloxyacetaldehydes. P. S. ARUP

1906. **Radio paper chromatography of isoniazid and its metabolites. I.** W. Diller, E. Krüger-Thiemer and E. Wempe (Allgem. Krankenhaus Heidberg, Hamburg-Langenhorn). *Arzneimittel-Forsch.*, 1959, **9** (7), 432–439.—Directions are given for the separation and identification of isoniazid and 14 related compounds by paper chromatography with *iso*propyl alcohol - 25% aq. NH_3 (17:3), *iso*propyl alcohol - H_2O (17:3) and *iso*propyl alcohol - formic acid - H_2O (8:1:1) as solvent systems. The compounds may be detected by u.v. light or by spraying with sodium nitroprusside, *p*-dimethylaminobenzaldehyde, 1:2-naphthaquinone-4-sulphonic acid, benzoyl chloride, ammoniacal $AgNO_3$ soln., or $CdSO_4$ soln., or the developed chromatograms may be scanned for radioactivity if labelled compounds are used. A. R. ROGERS

1907. **The identification of drugs. VI. Nitration of acetarsol.** K. Ilver, J. Berger and A. Jackerott (Lab. of the Pharmacopoeia Comm., Roy. Danish Sch. of Pharmacy, Copenhagen). *Acta Chem. Scand.*, 1959, **13** (5), 1046.—The nitration of acetarsol (0·1 g) with 5 ml of 2 *M* HNO_3 by boiling for 15 sec. and then cooling affords a cryst. yellow product, shown to be 2-acetamido-4:6-dinitrophenol (King, *J. Chem. Soc.*, 1927, 1049). The test is specific for acetarsol, since other arsenical drugs do not yield this compound. E. J. H. BIRCH

1908. **Gravimetric and infra-red spectrophotometric determination of piperazine.** W. R. Maynard, jun. (Div. Chem. Foods, Virginia Dept. Agric., Richmond, Va., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 610–612.—Piperazine, liberated from its salt with $NaHCO_3$, is acetylated in aq. $NaHCO_3$ soln. with acetic anhydride, and the diacetyl derivative, which is formed quantitatively, is extracted with $CHCl_3$. This soln. may be evaporated and the analysis completed gravimetrically, or the residue re-dissolved in $CHCl_3$ may be analysed spectrophotometrically at 10·03 μ. Results for standard samples agreed within 1%. A. A. ELDRIDGE

1909. **Non-aqueous titration of a new non-mercurial diuretic—chlorothiazide [6-chloro-7-sulphamoylbenzo-1:2:4-thiadiazine 1:1-dioxide].** Shu-Liang Chang. *Acta Pharm. Sinica*, 1959, **7** (8), 295–299.—*Procedure*—Transfer the sample (100 mg of pure compound or 150 mg of powdered tablet) to ethylenediamine (15 ml) containing *o*-nitroaniline indicator (5 drops) in a 50-ml conical flask. Close the flask with a rubber bung fitted with a 10-ml burette, a gas inlet and a gas outlet. Pass dried N into the flask and titrate with 0·2 *N* KOH in *iso*propyl alcohol until the colour changes from yellow to orange - red. The method gives a standard error of $\pm$0·4% for the pure compound and $\pm$0·47% for tablets. The sample may be satisfactorily titrated in acetone, azo violet being used as indicator. The end-point is sharp, but is not stable. Good results are also obtained when the sample is titrated potentiometrically in acetone, with antimony and calomel electrodes. S. H. YUEN

See also Abstracts—**1845**, Phenaglycodol in urine. **1846**, Oleandomycin in bile and urine.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1910. **Another comparison of a glucose oxidase method with an official method for the determination of fructose.** E. F. Potter, J. R. Wilson and K. T. Williams (Western Reg. Res. Lab., U.S. Dept. of Agric., Albany, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 650–652.—Results obtained for 46 vegetable samples by the A.O.A.C. method ("Methods of Analysis," 8th Ed., 1955, 29.64, 29.65) and by the method of Williams and Potter (*J. Ass. Off. Agric. Chem.*, 1958, **41**, 307, 681) do not vary in a regular manner. Though the methods are generally applicable, they may not measure precisely the same substances, and hence may not be expected to give identical values for all plant materials. A. A. ELDRIDGE

1911. **Standard analysis methods in the European confectionery industry.** C. Nieman. *Mfg Confect.*, 1959, **39** (6), 42, 44, 46–51.—The International Association of Confectionery Manufacturers has laid down standard methods of sampling and of analysis for refined sugar, lower grade sugar, invert sugar, glucose syrup, condensed milk, dried milk, fats, gelatin, agar, cocoa powder, pectin, tragacanth, essential oils and flavours, and finished goods. The methods are adapted from existing methods, to which reference is made. BRIT. BAKING IND. RES. ASS. ABSTR.

1912. **Catechins and catechin-tannins and their significance in foods.** K. Herrmann (Inst. f. biol. Forsch., Köln-Bayenthal, Germany). *Z. Lebensmittlntersuch.*, 1959, **109** (6), 487–507.—The paper-chromatographic method is used for detection and determination; *n*-butanol - acetic acid - water (4:1:5) and 15% acetic acid soln. are recommended as solvents. For two-dimensional work, the chromatogram is run first in *n*-butanol - acetic acid - water and then with 2% acetic acid soln. The following detecting reagents are used—(*i*) vanillin - HCl gives a red colour with catechins, phloroglucin and its derivatives and leucoanthocyanins, but not with flavones, flavonols and flavanones; (*ii*) $FeCl_3$ - $K_3Fe(CN)_6$ soln. gives a blue colour with polyhydric phenols; (*iii*) a mixture of equal parts of $AgNO_3$ soln. (2%) and aq. NH_3 (10%) gives a brown to black colour with all 1:2- and 1:4-dihydroxy- and 1:2:3-trihydroxy-benzene compounds; (*iv*) a soln. of $NaNO_2$ (1 g) in 2·5% methanolic acetic acid followed, after drying in air, with 0·5 *N* methanolic NaOH gives a red colour with many 1:2-dihydroxy-benzene compounds; (*v*) tungstophosphoric acid (Folin reagent) followed, after drying, with 0·5 *N* methanolic NaOH gives a blue colour with all 1:2- and 1:4-dihydroxy- and 1:2:3-trihydroxy-benzene compounds; (*vi*) $FeCl_3$ soln. (0·1 to 1·0%) gives green to blue colours with all 1:2-dihydroxy- and 1:2:3-trihydroxy-benzene compounds. The R_F values of catechins in various solvent mixtures are given. For quant. determinations the absorption spectrum in the u.v. is unsuitable, the max. for catechins occurring between 271 and 280 mμ, in which region most phenols absorb. S.C.I. Abstr.

1913. **Chlorinated solvents for determination of crude fat in feeds and meat products.** I. R. Sherman (Sherman Res. Lab., Toledo, Ohio, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 646–647.—Comparative tests with CCl_4, 1:1:1-trichloroethane and perchloroethylene on soya-bean-oil meal, maize, lucerne and meat products show that extraction is more complete than with diethyl ether, and that, since their use does not require previous drying of the sample, moisture can be determined simultaneously by use of a suitable measuring device. A. A. Eldridge

1914. **The distribution of acetic acid in fish-pickling baths and its determination by steam-distillation.** V. Meyer (Inst. für Meeresforsch., Bremerhaven). *Dtsch. LebensmittRdsch.*, 1959, **55** (8), 193–197.—Acetic acid diffuses not only into the tissue fluids, but also into the protein, whose buffering capacity interferes with direct titration when indicators are used. This interference can be eliminated by steam-distillation after acidification with phosphoric acid. The distribution of acid between fish and bath liquid is studied in relation to the ratio of bath liquid to fish weight, and the moisture, fat and protein contents of the fish. Empirical formulae are derived for calculation of final bath acidity. E. C. Apling

1915. **New reduction test for the examination of bottled milk.** J. Eisenbrand and A. Klauck (Chem. Untersuchungsamt, Saarbrücken). *Dtsch. LebensmittRdsch.*, 1959, **55** (7), 175–176.—A new colour test for the keeping quality of bottled milk, easily applied outside the laboratory, is described. A sterile 0·5% soln. of Brilliant black BN (C.I. Food Black 1) (2 ml per 500 ml of milk) is injected through the bottle cap by means of a hypodermic syringe. The puncture is closed with sterile plaster and the dye is mixed with the milk by repeated inversion. The bottle is kept at room temp. for 16 hr. and then examined. Decolorisation of the dye indicates poor keeping quality. E. C. Apling

1916. **Recovery of added monoglycerides from milk.** R. G. Jensen and G. W. Gander (Storrs Agric. Exp. Sta., Conn., U.S.A.). *J. Dairy Sci.*, 1959, **42** (7), 1235–1236.—Low recoveries of added monobutyrin and monostearin in the method described previously (*cf.* Jensen and Morgan, *Anal. Abstr.*, 1960, **7**, 264) are attributed to the presence of glycerol in the commercial monoglycerides. With purified compounds the recovery is quant. W. H. C. Shaw

1917. **Detection of intermediate compounds in the early stages of the browning reaction in milk products.** M. Keeney and R. Bassette (Maryland Univ., College Park, U.S.A.). *J. Dairy Sci.*, 1959, **42** (6), 945–960.—Methods are given for the determination of the free 5-hydroxymethylfurfuraldehyde (**I**) and of the **I** liberated from the products of the browning reaction of milk by treatment with oxalic acid under standardised conditions. After treatment with 2-thiobarbituric acid in the presence of trichloroacetic acid the resulting colour is measured at 443 mμ. The calibration curve for **I** is rectilinear from 1 to 3×10^{-5} *M*. Furfuraldehyde and methylfurfuraldehyde may be determined similarly. The significance of the results in relation to the development of off-flavours in milk products is discussed. W. H. C. Shaw

1918. **Simplification of standard methods for salt analysis in cheese.** G. J. Silverman, A. G. Wolin and F. V. Kosikowski (Cornell Univ., Ithaca, N.Y., U.S.A.). *J. Dairy Sci.*, 1959, **42** (6), 1095–1096.—Decantation or filtration of the pptd. AgCl in the A.O.A.C. method for the (Volhard) determination of NaCl in milk products is avoided by the use of nitrobenzene to protect the ppt. W. H. C. Shaw

1919. **Paper-chromatographic method for the detection of adulteration of tomato ketchup with sweet potato.** Om Prakash Beerh and G. S. Siddappa (Fruit Technol. Div., C.F.T.R.I., Mysore, India). *Food Sci., Mysore*, 1959, **8** (2), 45–46.—A circular paper-chromatographic method is described. In the presence of sweet potato a strongly defined band due to maltose is observed. N. E.

1920. **Characterisation of non-carbonyl volatiles of vanilla by gas chromatography.** H. P. Burchfield and E. A. Prill (Boyce Thompson Inst. for Plant Research, Yonkers, New York, U.S.A.). *Contr. Boyce Thompson Inst.*, 1959, **20** (3), 217–229.—Ethanolic extracts of vanilla were treated with 2:4-dinitrophenylhydrazine to precipitate carbonyl compounds, including vanillin, and were then extracted with *iso*pentane. After evaporation of the solvent, the residue was dissolved in dichloromethane, the solvent was again evaporated, and the residue was dissolved in acetone, and submitted to gas chromatography. Authentic vanilla extracts generally gave elution patterns with 4 characteristic peaks; in some, peak II (in order of increasing retention time) was double, and, in a few, peak III was double. Increasing the pH of the ethanol during extraction much increased the height of peak II. Beans from Tahiti gave patterns different from those of Mexican and Madagascan beans. Adulterations of commercial vanilla extracts that are not

revealed by paper chromatography can be detected by this method. M. D. ANDERSON

1921. **Collaborative study of the chromatographic evaluation of vanilla extracts.** D. Jorysch (H. Kohnstamm & Co., Inc., New York, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 638–642.—A collaborative assessment of the paper-chromatography method for the analysis of vanilla extracts (Burchfield *et al.*, *Amer. Perfum. Aromat.*, 1958, **71**, 49, 52) showed that it gives reproducible results. Solvent extraction removes interfering matter, *e.g.*, sugars; the outline of chromatographic patterns is not appreciably affected by time, temperature or alkali. A. A. ELDRIDGE

1922. **Separation and identification of food colours.** A. C. Cooke (Dominion Lab., D.S.I.R., Wellington). *N.Z.J. Sci.*, 1959, **2** (2), 260–265.—The 17 permitted water-sol. coal-tar food colours have been separated and identified by the paper-chromatographic method of Thaler and Sommer (*Z. LebensmittUntersuch.*, 1953, **97**, 345, 441) with 2% Na citrate soln. in 5% aq. NH_3 as developer, but modified by the use of *n*-butanol - 95% ethanol - water (1:1:1) as a second solvent. R_F values are tabulated for 41 food colours, including those authorised in England, and data are given for the identification of the spots. E. G. BRICKELL

1923. **Hydroxamic acids. IX. Micro-detection and determination of formic acid and inorganic formates. Application of the method to food products.** O. A. Guagnini and E. E. Vonesch (Dept. Org. and Biol. Chem., Buenos Aires). *An. Asoc. Quim. Argentina*, 1959, **47** (1), 41–51.—The method is based on the conversion of the acid into the ethyl ester, separation of the ester by distillation, its conversion into formohydroxamate, and, finally, detection and determination by the colour reaction with ferric perchlorate. A special micro-distillation apparatus is described for simultaneous esterification and distillation of the ester. The detection limit is 2 μg of formic acid and quantitative determinations of from 50 to 500 μg are possible with errors of <5%. Carboxylic acids, α-keto acids, aldehydes and reducing agents do not interfere. Results obtained with various food products vary from 0 to 45 mg of formic acid per 100 g. E. C. APLING

1924. **Detection of esters of *p*-hydroxybenzoic acid in wine.** F. Bucci and P. Tandoi (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1959, **22** (5), 495–507.—The method is based on the extraction of esters of *p*-hydroxybenzoic acid (**I**) with diethyl ether - light petroleum after treatment of the sample with $HgSO_4$ and NaOH soln. After evaporation of the solvent the residue is coupled with *p*-nitrobenzenediazonium chloride. The differentiation of the coloured azo compound derived from **I** from other coloured products which can interfere is based on the fact that the former can be extracted with $CHCl_3$ and dissolved in alkaline acetone to give a deep blue colour. This method can be used to detect **I** in concn. down to 10 p.p.m. The reaction is applicable to the identification of free *p*-hydroxybenzoic acid and its esters in foods. L. ZANONI

1925. **Determination of orange juice in beverages.** R. Intonti, F. Cotta-Ramusino and A. Stacchini (Ist. Sup. Sanità, Roma, Italy). *Chim. e Ind.*, 1959, **41** (7), 603–608.—The following determinations are proposed for orange juices and beverages prepared therefrom, detailed procedures being given—formol number, as a measure of the carboxyl groups of the amino acids, determined electrometrically; ninhydrin number, as a measure of the amino-acid content, determined spectrophotometrically; proline content, determined spectrophotometrically after reaction with ninhydrin; and arginine content, based on the spectrophotometric measurement of the colour produced with 1-naphthol and bromosuccinimide. L. A. O'NEILL

1926. **Fluorimetric determination of quinine in tonic water.** F. Strache (Chem. u. Lebensmitt. Untersuchungsanst. Hygien. Inst., Hamburg, Germany). *Dtsch. LebensmittRdsch.*, 1959, **55** (6), 153–154.—The method depends on the fluorescence of quinine sulphate in H_2SO_4 soln. The fluorescence is diminished by the addition of Cl^- as NaCl or HCl, and intensified by the addition of H_2SO_4. For a 1% soln. of H_2SO_4 the increase is 9%. The addition of 35·5 mg of Cl^- per litre gives a decrease of 10%. This error can be corrected by reference to curves or tables for known Cl^- concn. No samples of tonic water tested contained Cl^- >0·0004 *N*. The fluorescence was measured in the concn. range equiv. to 1 to 10 mg of quinine per litre in an Eppendorf photometer, and referred to a quinine soln. containing 10 mg of anhyd. quinine in 1 litre of 4·5% H_2SO_4. The addition of the same quantities of NaCl for all quinine concn. gave the same decrease of fluorescence. *Procedure for lemonade* (*containing <14 mg of Cl^- per litre*)—The reference soln. is prepared by dissolving quinine sulphate (115·1 mg = 100 mg of quinine) in a little dil. H_2SO_4 and making up with water to 1 litre. For use, an aliquot (10 ml) is made up to 100 ml with 5% H_2SO_4 soln. The sample (10 ml) is made up to 100 ml with 5% H_2SO_4. The instrument is set at 100% transmission for the reference soln.; the transmission of the sample soln. is then measured, and corrected for Cl^- concn. by the table given. S. C. I. ABSTR.

1927. **Conductimetric determination of α-acids in green hops on the hop farm.** B. D. Hartong and L. Isebaert. *Wallerstein Labs Commun.*, 1959, **22**, 17–26.—The method described is very accurate and can be carried out in 15 to 20 min. For the determination of α-acids in normal dried hops, 15 g is placed in a blender with 100 ml of pure benzene (or 7·5 g of hops with 50 ml of benzene). After a 5-min. extraction, 10 ml of the unfiltered supernatant soln. is pipetted into the beaker of the titration assembly, which contains 40 ml of methanol (methanol denatured with acetone or benzene is suitable). The soln. is titrated with a clear 4% methanolic soln. of Pb acetate. The assembly may consist of a dip cell and resistance meter or a conductivity meter. Lead acetate soln. is added in 0·5-ml aliquots, preferably with magnetic stirring, and resistance readings are made after each addition. As soon as the resistance falls, the additions of Pb acetate are reduced to 0·1 ml. The results are plotted, and it is observed that there is no change in conductivity as long as the α-acids are being pptd. When pptn. is complete, the conductivity increases rapidly and the curve shows a sharp inflection; thereafter a steeply rising straight line is obtained; the point of intersection of the two straight lines produced gives the vol. of Pb acetate required for complete pptn. The ml of Pb acetate × 2·54 (for humulone) gives the figure for α-acids. With green hops, 30 g is required because of the high moisture

content and 1% Pb acetate soln. is used for the titration. In making calculations a factor of 0·318 is then used. J. Inst. Brew. Abstr.

1928. **Copper and iron determination in hops.** H. E. Jansen, M. J. M. M. Luykx, B. D. Hartong, W. J. Klopper and C. G. T. P. Schnellen. *Int. Tijdschr. Brouw. Mout.*, 1958–1959, **18**, 158–162.—Six laboratories have carried out a collaborative study of some of the methods for determining Fe and Cu in beer. For Fe, Gray and Stone's direct method was used, comparing 2:2′-dipyridyl (**I**) and 1:10-phenanthroline (**II**) as the final colorimetric reagents. Ferrous ammonium sulphate was used for the preparation of standard curves. Both reagents gave results which showed good agreement between laboratories, but the results with **I** were influenced by the reaction temp. whereas the colour developed with **II** was not (20° and 60° were used); **II** is therefore recommended. In preliminary experiments for the determination of Cu, several methods of wet-ashing were used by some of the laboratories, and the Cu was then determined colorimetrically by means of Na diethyldithiocarbamate (**III**); in two other laboratories the direct Zn dibenzyldithiocarbamate (**IV**) method of Stone and the Hartong direct **III** method were used. A wide spread of results was obtained. In further experiments, the ashing - **III** procedure was compared with the direct **IV** method. The former gave the more uniform results, and was therefore preferred; the ashing procedures recommended were those employing HNO_3 and H_2SO_4 or H_2SO_4 and $HClO_4$. J. Inst. Brew. Abstr.

1929. **Studies on the non-biological hazes of beers. VIII. Rapid estimation of anthocyanogens in beer.** G. Harris and R. W. Ricketts (Brewing Ind. Res. Found., Nutfield, Surrey, England). *J. Inst. Brew.*, 1959, **65** (4), 331–333.—De-gassed beer (10 ml) is shaken with nylon-66 powder (0·5 g) for 40 min. The resin is filtered off, washed with water and heated at 100° with *n*-butanol - HCl (5:1) (**I**). After stirring at intervals of 1 and 3 min. a clear soln. is obtained which is heated for a further 30 min. After cooling, the red - purple soln. is diluted to 25 ml with **I** and the extinction is measured at 550 mμ against a blank prepared by treating fresh nylon in the same way. A comparison of the colours obtained from beers with those formed under the same conditions by authentic leucocyanidins shows that it is possible to relate the colour from any beer directly with the weight of an authentic anthocyanogen taken as an arbitrary standard. The method is accurate and reproducible. S. M. Marsh

1930. **Determination of potassium and sodium in wine. Comparison of flame-photometric with gravimetric methods.** W. Diemair and C. Gundermann (Univ.-Inst. Lebensmittelchem., Frankfurt a.M., Germany). *Z. LebensmittUntersuch.*, 1959, **109** (6), 469–474.—Determinations of K and Na in 25 wines were made by flame photometry and compared with gravimetric determinations by known methods. The former method has many advantages and gives good reproducibility. S.C.I. Abstr.

1931. **The analytical chemistry of wine. VI. Detection and determination of titanium [and iron] in wine with tiron.** H. Eschnauer (Anorg.-Chem. Inst., Johannes-Gutenberg Univ., Mainz, Germany). *Z. LebensmittUntersuch.*, 1959, **109** (6), 474–479.—*Procedure*—The sample (50 ml) is decomposed with HNO_3 and H_2SO_4 in a platinum dish, and the residue is dissolved in 3 ml of 2 *N* H_2SO_4 with warming. The liquid is filtered into a 25-ml flask, and the residue is washed twice with 1 ml of water. *Determination of Ti*—The soln. in the 25-ml flask is treated with 3 ml of 4% aq. tiron soln. (**I**), then with 2 *N* aq. NH_3, dropwise, till the colour of Congo red paper changes from red to blue. Acetate buffer soln. (pH 4·7) (5 ml) is added, the mixture is made up to vol. and shaken, and the pH adjusted to 4·7 (pH meter); $Na_2S_2O_4$ (**II**) (25 mg) is then added, and dissolved with the least possible stirring, and the yellow colour is measured at 410 mμ within 5 min. against a reagent blank containing the same amount of neutral salts as the sample soln. *Determination of Fe and Ti*—The blue - violet colour of the soln. at pH 4·7, obtained as described for the determination of Ti, is measured at 560 mμ; **II** is then added as described above, and the yellow colour measured at 410 mμ. Results are referred to calibration curves. Aluminium, As, B, Be, Br, Cd, Cr, Cu, Cl, I, Mg, Mn, Mo, U, W and Zn do not interfere; V, if present in amount greater than that of the Ti, must be removed. Titanium ranged from 0·04 to 0·23 mg per litre in the 35 wines examined. S.C.I. Abstr.

1932. **An improved ashing technique for the determination of lead in wines and spirits.** J. P. van der Westhuyzen (Div. of Chem. Services, Cape Town, S. Africa). *S. Afr. J. Agric. Sci.*, 1959, **2** (2), 183–186.—The sample (10 ml) is placed in a silica basin with 5 ml of "ash-acid" soln. (25 g of anhyd. Na_2SO_4 dissolved in 500 ml of 10% HNO_3), and evaporated to half its volume on a water bath. Conc. HNO_3 (3 ml) and a little cold water (redistilled in all-Pyrex apparatus) are added and the mixture is evaporated to dryness on the water bath, further dried and charred and then ashed at 450° for 15 min. After cooling, the residue is moistened with conc. HNO_3, dried and re-ashed, repeating this procedure if necessary. The ash is washed into a Pyrex-glass separating-funnel with 25 ml of hot dil. HNO_3 (1%), 5 ml of hot ammonium acetate soln. (20%) and 25 ml of cold dil. HNO_3 (1%), and after the addition of 1 ml of hydroxyammonium chloride soln. (1%), 2 ml of citric acid soln. (50%) and four to six drops of thymol blue indicator (0·1%) the contents of the funnel are neutralised with conc. aq. NH_3. Potassium cyanide soln. (10%) (1 ml) and a soln. of dithizone in $CHCl_3$ (0·0015%) (20 ml) are added and the Pb is determined colorimetrically. The recovery of Pb is complete for dry wines and spirits, and ≃88% for sweet wines. The procedure is accurate and reproducible, the time required is considerably less than that for the usual wet-ashing procedure, and no interfering elements are introduced. S. M. Marsh

1933. **The use of the gas chromatograph in the determination of fusel oil in grape brandy.** R. J. Bouthilet and W. Lowrey (E. & J. Galls Winery, Modesto, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 634–637.—By the use of a Beckman Flexol column at 130° and a Beckman GC-2 gas chromatograph, good separation of fusel-oil components was achieved. The results obtained by this method were, however, not in accord with those obtained by the colorimetric procedure employed for comparison (Penniman *et al.*, *Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 991). A. A. Eldridge

1934. **[Modified] cold process of saponification and its advantages.** A. K. Mallik, S. N. Mitra and T. V. Mathew (Central Food Lab., Calcutta). *J. Instn Chem., India*, 1959, **31** (3), 122–123.—*Procedure*—The sample (5 g) is mixed with 0·5 *N* ethanolic KOH (50 ml) and the mixture is heated for 5 min. on a steam bath without boiling, then cooled to room temp. and light petroleum (boiling-range, 40° to 60°) (5 ml) is added. The contents are mixed and the flask is set aside overnight. The excess of alkali is then titrated with 0·5 *N* HCl. Results agree with those by the official A.O.A.C. method.
C. A. SLATER

1935. **Use of infra-red spectrography in the field of fats and oils. I. Literature.** H. P. Kaufmann, F. Volbert and G. Mankel (Dtsch. Inst. für Fettforsch., Münster, Westphalia). *Fette, Seif., Anstrichmitt.*, 1959, **61** (7), 547–559.—A review, with 249 references.
P. D. PARR-RICHARD

1936. **Paper chromatography in the field of fats and oils. XXX. Analysis of glycerides.** H. P. Kaufmann and H. Schnurbusch (Inst. für Pharm. u. Lebensmittelchem. der Univ. Münster, Westphalia). *Fette, Seif., Anstrichmitt.*, 1959, **61** (7), 523–528.—For the separation of soya-bean-oil glycerides, silicone or liquid paraffin was used as the stationary phase and acetone - methyl cyanide as the mobile phase. *Procedure*—Dissolve 2 g of oil in benzene and purify by passing it through 5 g of Al_2O_3, collecting the eluate under N. Impregnate the paper with liquid paraffin (10% soln. in light petroleum, boiling-range 60° to 90°) and press between paper to dry. Take 60 μg of oil in benzene for the test and develop with acetone - methyl cyanide (3:1). After 5 hr. dry at 80° for 15 min.; the spots are detected by exposure to iodine vapour. Individual glycerides are identified by saponification and separation of the fatty acids, which are converted into the copper soaps and treated with $K_4Fe(CN)_6$; the depth of colour is measured photometrically. The glyceride composition of the re-esterified oil was appreciably different from that of the natural product.
P. D. PARR-RICHARD

1937. **Separation of fatty-acid esters by gas chromatography.** A. Jart (Dansk Fedtforskningsinst., Copenhagen). *Fette, Seif., Anstrichmitt.*, 1959, **61** (7), 541–546.—Fatty acids are converted into their methyl esters for analysis. The column (illustrated) consists of 2 stainless-steel tubes (i.d. 8 mm, total length 2 metres); the packing is of silicone grease supported on steel Dixon rings ($\frac{1}{16}$ in. × $\frac{1}{16}$ in.). Two katharometers of 22 ohms resistance form the arms of a Wheatstone bridge; a current of 85 mA is passed and the curve is recorded by a 5-mV Philips potentiometer. After passing through the packed column and past one katharometer, the esters are separated by freezing from the carrier gas (N), which is then reheated in an unpacked section of the column and finally flows past the second detector. Traces of unsaturated acids are detected by broadening of the curves, but for separation of mixtures of unsaturated acids a Celite - adipic polyester packing is preferable. The separation of different acid mixtures and the theoretical aspects of quant. determination are discussed.
P. D. PARR-RICHARD

1938. **Review of the analysis of olive oil, with particular regard to colour reactions.** S. Anselmi (Ist. Sup. di Sanità, Roma). *Olii Min.*, 1959, **36** (6), 210–213.—Methods for detecting adulteration in olive oil are critically reviewed, and conditions for the Fitelson test (*J. Ass. Off. Agric. Chem.*, 1936, **19**, 493) for teaseed oil in olive oil are discussed.
L. A. O'NEILL

1939. **Differentiation of olive oils classified as 'rectified B.'** V. Sossi (R.I.O., Imperia, Italy). *Olii Min.*, 1959, **36** (6), 214–216.—The chemical constants, flavour and behaviour in various reactions of 10 oils classed as "rectified B" are tabulated. Oils prepared by esterification of the distilled fatty acids obtained from neutralisation pastes of olive or sulphur olive oils had characteristics similar to those of the classical "rectified B" oils obtained by neutralisation of extracted olive oil. Oils from other sources showed clear distinctions.
L. A. O'NEILL

1940. **Detection of grape-seed oil in olive oil by paper chromatography.** G. Petruccioli (Ist. Sper. per l'Olivicoltura, Spoleto, Italy). *Olii Min.*, 1959, **36** (7), 260–264.—The presence of small amounts (5%) of grape-seed oil in olive oil (expressed or "rectified B") cannot be established with certainty from the analytical constants. A chromatographic procedure is described, based on the higher content of polyunsaturated acids in grape-seed oil, that involves an initial fractionation of the fatty acids on an alumina column, followed by a one-dimensional separation on paper.
L. A. O'NEILL

1941. **Detection of small amounts of castor oil in vegetable or animal oil.** S. Anselmi, L. Boniforti and R. Monacelli (Ist. Sup. San., Roma). *Olii Min.*, 1959, **36** (7), 253–255.—The oil, to which has been added an equal quantity of paraffin wax, is extracted with hot ethanol (90°), the alcohol is removed, and the oily residue purified from free fatty acids. Castor oil present is identified by the formation of a red - orange product on condensation with *p*-dimethylaminobenzaldehyde, which is extractable with $CHCl_3$ and has an absorption maximum at 532 mμ. An estimate of the amount of castor oil present can be obtained spectrophotometrically; alternatively, this may be done gravimetrically by carrying out the extraction and purification under controlled conditions.
L. A. O'NEILL

1942. **Gas - liquid chromatography of fatty derivatives. II. Analysis of fatty alcohol mixtures by gas - liquid chromatography.** W. E. Link, H. M. Hickman and R. A. Morrissette (Archer-Daniels-Midland Co., Minneapolis, Minn., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1959, **36** (7), 300–303.—Fatty alcohols are converted into their acetates and applied in 2-μl quantities to a 9-ft. chromatographic column maintained at 203° to 230°, with He as carrier at a flow rate of 42 ml per min. The column is packed with an adsorbent mixture consisting of 15% of a polyester (Resoflex 446) on a support of Chromosorb W (60 to 80 mesh). The relationship between peak areas and wt. (%) of four C_{18} alcohols from linseed oil is discussed. Fatty alcohols derived from soya-bean, linseed and sperm oils have been prepared and analysed.
G. R. WHALLEY

1943. **Detection of peroxides on filter-paper.** T. Patzek (Inst. f. NE-Metalle, Gliwice, Poland). *Chem. Tech., Berlin*, 1959, **11** (6), 326–327.—The method developed for histological purposes by Dubouloz and Dumas (*C.R. Soc. Biol., Paris*, 1954, **148**, 5, 551), which depends on the oxidation of Cu^+ to Cu^{2+} and the formation of a complex compound

with dithio-oxamide, is also a sensitive and simple method for the detection of peroxides in fats and solvents. Pure CuCl is added to a 6% aq. soln. of KSCN, shaken for 10 min. and filtered. Immediately before use, 0·4 ml of 0·25% ethanolic dithio-oxamide soln. is added to 10 ml of the filtrate. The sample is dissolved in a volatile solvent (free from peroxides) and one or two drops are put on a strip of filter-paper. When the solvent has evaporated the paper is immersed in the freshly prepared reagent for 10 min.; an olive-green spot appears, the intensity of which depends on the amount of peroxides present. The colour is stable for a long time if protected from light. I. DICKINSON

1944. **Analysis of fat-soluble vitamins. IV. Discussion of the routine chemical determination of vitamin D.** K. H. Hanewald (Anal. Dept., N.V. Philips-Roxane, Pharm. Chem. Ind. "Duphar," Weesp, The Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1959, **78** (8), 604–621 (in English).—Ergocalciferol and cholecalciferol in irradiation products of ergosterol and 7-dehydrocholesterol were determined spectrophotometrically at 500 mμ, antimony trichloride and acetyl chloride in $CHCl_3$ being used as the colour reagent. Tachysterols were eliminated by addition of maleic anhydride at 75°, provitamins and other sterols by precipitation with digitonin, and oils by saponification. Two procedures for complete determination are suggested.
M. H. SAWISTOWSKA

1945. **Anorganic bone as a measure of calcification in the A.O.A.C. vitamin-D assay.** F. D. Wharton, jun., I. Olcott, L. J. Classen, R. E. Apple and J. C. Fritz (Dawe's Laboratories, Inc., Chicago, Ill., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 623–624.—The percentage of ash in dry, fat-extracted chick tibiae is ≃7 times (average 7·08 ± 0·85) the percentage of residue remaining after extraction of organic matter from the fresh material with ethylenediamine for 30 hr. The same response curve was obtained by plotting the data on suitable scales. The use of ethylenediamine is an acceptable alternative procedure to dry-ashing, but the accuracy of the assay is not thereby improved. A. A. ELDRIDGE

1946. **Chromatographic separation and fluorimetric measurement of vitamin-B6 components in aqueous solutions.** M. J. MacArthur and J. Lehmann (Human Nutrition Res. Div., Agric. Res. Service, U.S. Dept. Agric., Washington, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 619–622.—The resin Dowex AG 50-WX 8 (sodium form) permits complete separation of pyridoxal, pyridoxol and pyridoxamine, which are eluted respectively with hot soln. of K acetate acidified with acetic acid (0·04 *M*, pH 5·5; 0·05 *M*, pH 6·1) and K citrate (0·2 *M*). Recoveries of 98%, 99% and 99% were achieved. Procedures for oxidation of these substances to 4-pyridoxic acid and conversion into the lactone (Fujita *et al.*, *J. Vitaminol.*, 1955, **1**, 267, 275, 279, 290) give relatively reproducible but not quantitative results when the lactone is determined spectrophotofluorimetrically. A. A. ELDRIDGE

1947. **Modified procedure for the determination of niacin [nicotinic acid] in cereal products.** O. Pelletier and J. A. Campbell (Food and Drug Lab., Dept. Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 625–630.—The official (A.O.A.C.) method is improved by using 1·5% $Ca(OH)_2$ soln. instead of H_2SO_4 for the digestion. Colour is developed by reaction with cyanogen bromide at 2°; max. extinction occurs at 470 mμ (A.O.A.C. method, 435 mμ). Recoveries were 96 to 103% (A.O.A.C. method, 74 to 110%). The modified procedure gives lower blank readings and is subject to less interference by extraneous substances. A. A. ELDRIDGE

1948. **Effect of water on the determination of tocopherols.** A. Hobson-Frohock (Dunn Nutritional Lab., Milton Rd., Cambridge, England). *Analyst*, 1959, **84**, 567–568.—Since the chromatographic paper alone gives a measurable extinction with the 2:2′-dipyridyl - $FeCl_3$ reagent, a blank sheet must be included with the test sheets in the separation and determination of tocopherols. Also, since the extinction is affected by the presence of water in the ethanolic dipyridyl - $FeCl_3$ reagent, the blank and test papers must be dried to the same extent. A simple procedure is to spin the metal frame containing the blank and test papers on a turn-table at 170 r.p.m. in air in dim light for 6 min. By this method the mean extinction of the blank soln. from 50 determinations was 0·065 ± 0·0046.
A. O. JONES

See also Abstracts—**1721,** SO_2 in fruit juice and cider. **1792.** Determination of sorbic acid. **1875,** 5-Vinyl-2-oxazolidinethione in milk. **1889,** Detection of opium in foods. **1974,** Dyrene in vegetables.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1949. **Method for measuring the moisture content of air.** J. Higgins (Mining Res. Estab., Nat. Coal Board, Isleworth, Middx., England). *J. Sci. Instrum.*, 1959, **36** (7), 300–302.—The water vapour in the air sample contained in a flask is condensed as ice, the dry air is removed by pumping, and the pressure produced on re-evaporation of the ice in a closed evacuated space is measured. The apparatus described enables absolute humidities to be determined with an accuracy within 1%.
G. SKIRROW

1950. **Determination of traces of sulphur in small amounts of air.** H. Zimmermann (Inst. of Hygiene of the Ruhr, Gelsenkirchen, Germany). *Angew. Chem.*, 1959, **71** (10), 336–338.—The sensitivity of Wickbold's method for the turbidimetric micro-determination of SO_4^{2-} (*Ibid.*, 1953, **65**, 159) has been increased 200-fold so that 1·0 μg of S can now be accurately determined. The volume of the test soln. was reduced by using a cell with 20-mm transmission, but only 3 mm wide; with a 10-mm cell, 0·5 μg can be determined less accurately. A very sensitive Eppendorf photometer was used. During the addition of the titrant (0·0191% aq. $BaCl_2$ soln.), the soln. should be stirred mechanically. The electrolyte is a soln. of $MgCl_2$ (20 g) and NH_4Cl (2·5 g) in H_2O (1 litre), adjusted to pH 8·0 to 8·2 with aq. NH_3. To decrease the volatility of SO_4^{2-} when evaporating a soln., add a 1% aq. soln. of NaCl. The sensitivity can be increased by adding an aq. soln. of K_2SO_4 (0·543 g, or less, per litre) and deducting the amount of S thus added from the value obtained. The apparatus for sampling the air and oxidising the sulphur compounds is described. The accuracy is a few per cent. in the determination of 0·5 μg of S; by adding SO_4^{2-}, 0·1 μg of S (*i.e.*, 0·2 p.p.m. of S in a few litres of air) can be determined with lower accuracy. A. G. PETO

1951. **Detection of trace constituents by gas chromatography. Analysis of polluted atmosphere.** P. S. Farrington, R. L. Pecsok, R. L. Meeker and T. J. Olson (Dept. of Chem., Univ. of Calif., Los Angeles, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1512–1516.—Techniques are described for the detection and quant. determination of components present in the atmosphere at concn. of a few parts per 10^8, in the presence of a comparatively large amount of water vapour. The methods are applicable to a large variety of organic compounds, including saturated and unsaturated hydrocarbons, aromatics and most oxygenated materials likely to be present in the atmosphere. Quant. determinations are made by measuring peak areas. At levels of a few parts per 10^8 the estimated error is $<50\%$.

K. A. PROCTOR

1952. **Determination of the total hardness of water.** E. Luquin (Fac. de Quím. y Farm., La Plata, Argentina). *Monit. Farm. Terap.*, 1959, **65**, 363–365.—Modifications have been made to the method of Wartha-Pfeiffer (*Z. angew. Chem.*, 1902, **15**, 193). Known amounts of $CaCl_2$ and $MgCl_2$ are added to a duplicate sample to act as internal standards. The results are more precise, and between 2% and 4% higher, than those obtained by the Wartha-Pfeiffer method. The use of Xylene Cyanol FF (C.I. Acid Blue 147) to screen the methyl orange indicator interferes with the end-point if the solution is boiled. By omitting the pH adjustment of the distilled water used in the blank titration better results are obtained. T. R. MANLEY

1953. **Determination of chloride by precision null-point potentiometry.** H. V. Malmstadt and J. D. Winefordner (Univ. Illinois, Urbana, Ill.). *J. Amer. Wat. Wks Ass.*, 1959, **51** (6), 733–741.—The apparatus and procedure are described for the rapid and accurate determination of Cl^- in samples of ground and river water. Small samples (0·01 to 10 ml) are used to determine Cl^- concn. from 4000 to 0·04 mg per litre. The entire procedure occupies approx. 1 min. Interfering ions include I^-, Br^-, CN^-, S^- and Fe^{2+}. O. M. WHITTON

1954. **Determination of bromide and iodide in natural waters.** Yasumitsu Uzumasa, Masakichi Nishimura and Yoshikazu Nasu (Chem. Dept., Fac. of Sci., Hokkaido Univ., Sapporo). *Japan Analyst*, 1959, **8** (4), 231–234.—Kolthoff's method (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 75) can be satisfactorily applied to the determination of the sum of Br^- and I^- in natural waters, including hot springs and oil-well brine, provided that the concn. of each ion is increased to $>0·5$ mg per litre by evaporation in the presence of excess of Na_2CO_3. The amount of I^- is determined by Winkler's method (*Z. angew. Chem.*, 1915, **28**, 494). The presence of Cl^- (>5000 times the wt. of the Br^-) and a normal amount of common ions does not cause interference.

K. SAITO

1955. **Improvements in apparatus for the measurement of dissolved oxygen.** Cambridge Instrument Co., Ltd. [Inventor: G. Jessop]. Brit. Pat. 818,732; date appl. 15.2.57.—The apparatus comprises a scrubbing tower for removal of the dissolved oxygen (*e.g.*, from a sample of boiler feed water) by scrubbing with an inert gas. An electro-chemical cell, for measuring the concentration of O in the inert gas, comprises an anode consisting of a platinum wire coated with platinum black and a hydrogen-saturated solution of an electrolyte, the pH of which is so adjusted that, when there is no O present in the gas stream entering the cell, the cathode is polarised. When O is admitted into the cell it combines with H^+ round the cathode to form water, liberating 4 positive charges for each molecule of O. The resulting current in the external circuit is proportional to the concn. of O in the electrolyte, and serves as a measure of the O content of the sample. J. M. JACOBS

1956. **Concentration by electrodialysis of caesium and strontium radioactivities in water.** M. J. Owers [U.K.A.E.A. (Res. Group), C.37, Royal Arsenal, Woolwich, England]. A.E.R.E. Report AERE-R 3010, 1959, 11 pp.—The sample ($\simeq 100$ litres) containing caesium and strontium carriers is placed in an earthenware pot in which there is a cylindrical cathode compartment (vol. $\simeq 1$ litre) made from a membrane of Permaplex C10 ion-exchange resin. Owing to the transfer of Ca with the Cs and Sr when hard tap-waters are treated it is necessary to fill the cathode compartment with 3 *M* HNO_3 before starting the electrodialysis. Recoveries of at least 80% for Cs and Sr are possible with two successive treatments, each of 6 hr., and with a current of 10 amp. The apparatus is illustrated and details of the circuit are given.

G. J. HUNTER

1957. **Determination of alkylbenzenesulphonates in river water and sewage.** H. L. Webster and J. Halliday (Chem. Div., Thomas Hedley & Co. Ltd., P.O. Box 155, City Road, Newcastle upon Tyne, England). *Analyst*, 1959, **84**, 552–559.—The sample containing the anionic detergent is hydrolysed with dil. H_2SO_4, neutralised, and extracted with $CHCl_3$ containing 1-methylheptylamine. The extract is evaporated, the residue is dissolved in water and the soln. is extracted with light petroleum containing 1-methylheptylamine. After removal of the methylheptylamine, the detergent - methylene blue complex is formed in $CHCl_3$ soln., and its extinction is measured at 650 mμ or absorptiometrically with suitable filters. By means of calibration graphs the extinction is referred to concn. of alkylbenzenesulphonate or Manoxol O.T. [di-(2-ethylhexyl) sodium-sulphosuccinate]. The method gives good results with known amounts of detergent, and is reproducible. A. O. JONES

1958. **Oxidised nitrogen in water and sewage effluents observed by ultra-violet spectrophotometry.** R. C. Hoather and R. F. Rackham (The Counties Public Health Lab., 66 Victoria St., London). *Analyst*, 1959, **84**, 548–551.—The specific absorption coeff. at 210 mμ for nitrate (1 p.p.m. of N) is 0·55. For nitrite, the corresponding figure is 0·37, but nitrites usually occur only in small traces. Correction for organic matter can be applied on the empirical basis that the extinction of organic matter at 210 mμ is four times that at 275 mμ, at which wavelength the extinction of NO_3^- is negligible. Results with waters of moderate organic purity agree with those of other methods. With some samples containing NO_3^- freshly formed by microbiological action, discrepancies suggest the presence of unstable compounds (possibly of the form $Na_2N_2O_2$) that change slowly to nitrate. The method is suggested as an additional means of investigating nitrification in soils, water and sewage effluents.

A. O. JONES

1959. **Micro-determination of dissolved oxygen in boiler waters by constant-current coulometry. I.**

J. Hissel and J. Pire (Cebedeau, 2 rue Armand Stévart, Liège, Belgium). *Bull. Cent. Belge Étud. et Docum. Eaux*, 1959, (44), 76–86.—In the procedure described, $MnSO_4$, KI and an excess of 0·001 *N* $Na_2S_2O_3$ are added to the sample and to a blank. Iodine is then generated coulometrically to titrate the excess of $Na_2S_2O_3$. The end-point is determined amperometrically. No standard soln. are necessary, as the soln. are standardised electrically.

II. Description of micro-analyser for oxygen. R. Massart, M. Schutz and J. Hissel. *Ibid.*, 1959, (44), 86–88.—Apparatus developed for the coulometric determination of dissolved oxygen in boiler water (*cf.* Hissell and Pire, Part I, above) is described. J. H. WATON

See also Abstracts—**1677**, Determination of ^{89}Sr and ^{90}Sr in water. **1695**, Silica in water. **1698**, Lead and Cu in water. **1742**, Chloride in water. **1781**, Dichlorodifluoromethane in fumigation mixtures.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1960. **Capture of γ-radiation from cadmium as a means of slow-neutron detection in the determination of soil moisture.** V. A. Emel'yanov and V. E. Nesterov. *Atomnaya Energiya*, 1959, **6** (5), 573–575.—Two field methods of measuring soil moisture, based on the retardation of fast neutrons by water, are compared. The well-known proportional boron counter method is compared with a so-called "cadmium difference" method: γ-radiation is measured with and without a cadmium screen and the γ-radiation captured from the cadmium screen ("cadmium difference") is proportional to the neutron density at the place of measurement. A halogen-filled counter is fitted with a demountable screen of sheet cadmium (0·5 mm thick); Po - Be used as the fast-neutron source ($\simeq 1 \times 10^6$ neutrons per sec.) is housed in a cylindrical lead shield. Impulses are registered on the usual low-gauge battery radiometer with a thyratron conversion circuit with a cold cathode. Tests are carried out in cylindrical tanks (diam. and height 30 cm) charged with artificial soil mixtures; the detector tube is inserted into 5-cm access holes. A proportional boron counter is used as the control. The relation (curvilinear) between the percentage (vol.) of water in the soil and the meter reading is the same whichever method is used, but the "cadmium difference" method is much simpler and the sensitivity to moisture changes is greater. Preliminary tests indicate that the method can be used for clay, sandy soils, loam, sand and gravel. K. R. C.

1961. **A modified activity-index procedure for determining the quality of the water-insoluble nitrogen in mixed fertilisers containing urea - formaldehyde compounds.** K. G. Clark, J. Y. Yee, F. O. Lundstrom and T. G. Lamont (Fertiliser Invest. Res. Branch, Agric. Res. Service, U.S. Dept. of Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 592–597.—Values for a series of fertiliser materials and fertiliser mixtures are tabulated as follows—total N, pH, cold- and hot-water-insol. N, neutral permanganate insol. N, degree of nitrification in soil in 3 to 15 weeks, nitrification index, neutral permanganate index, and activity index. With one exception the modified procedure (involving a change in the quantity of sample and buffer solution) correctly evaluated the nitrification characteristics of the mixture. A. A. ELDRIDGE

1962. **The direct determination of available phosphorus in fertilisers by the quinolinium molybdophosphate method.** C. H. Perrin (Canada Packers Ltd., Toronto, Canada). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 567–568.—The quinolinium molybdophosphate method for the gravimetric determination of available P (Perrin, *Ibid.*, 1958, **41**, 758) has been applied in a simplified form to fertilisers and to cottonseed meal with satisfactory results. Unless available P in inorganic form is present, the ammonium citrate need not be destroyed. A. A. ELDRIDGE

1963. **Spectrophotometric method for direct available phosphoric oxide in fertilisers.** C. W. Gehrke and F. J. Johnson (Dept. of Agric. Chem., Univ. of Missouri, Columbia, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 569–574.—The method, which is fully described, involves extraction of the available P_2O_5 with water and then with aq. ammonium citrate soln., removal of organic matter (including citrate) and hydrolysis of metaphosphate by digestion of the extract with a mixture of HNO_3, $HClO_4$ and H_2SO_4, development of colour by the official (A.O.A.C.) molybdovanadate method for total P, and spectrophotometric determination of P at 400 mμ. The method is rapid, precise and accurate, the accuracy being comparable with that afforded by the A.O.A.C. photometric and alkalimetric methods. A. A. ELDRIDGE

1964. **Direct determination of available phosphorus in fertilisers: use of sodium chlorate to destroy citrate.** J. A. Brabson and W. D. Wilhide (Div. Chem. Development, Tennessee Valley Authority, Wilson Dam, Ala., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 574–578.—The combined aqueous extract and ammonium citrate solution extract is heated with $NaClO_3$ solution and HNO_3; if organic phosphorus is present a second oxidation, followed by heating to fuming with $HClO_4$, is necessary. The P is then determined volumetrically or spectrophotometrically. The saving in time is particularly marked if the latter procedure is employed. A. A. ELDRIDGE

1965. **A water problem in sampling and analysis of triple superphosphate.** W. L. Hill, J. Y. Yee and H. P. Freeman (Fertiliser Invest. Res. Branch, Agric. Res. Service, U.S. Dept. of Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 598–606.—Samples of triple superphosphate vary considerably in their capacity to absorb water from the ambient atmosphere, high-grade products exhibiting high avidity. An extensive study has been made of sorption in relation to humidity and time of exposure of granular and powdered samples, and results are presented in tables and graphs. Precautions necessary in sampling, grinding and weighing are considered. A. A. ELDRIDGE

1966. **Preparation of a complex fertiliser by treating rock-phosphate with nitric acid. III. Analytical methods.** A. Tancsa. *Nehézvegyipari Kutató Intézet Közleményei*, 1959, **1** (3–4), 183–190.—Two rapid procedures are given for the determination of P_2O_5 and CaO in soln. used for the preparation of the fertiliser. (*i*) A modification of Neuberger's amperometric method permits P_2O_5 to be titrated in the presence of Fe^{3+}. There is a high negative error

caused by Ca^{2+}, increasing to a constant value of −3·1% as the ratio of CaO to P_2O_5 increases to 8; (*ii*) CaO is determined by adding an excess of EDTA to the neutral soln. and back-titrating the excess with a standard soln. of $ZnCl_2$, with Eriochrome black T or (if Fe^{3+} or Al^{3+}, or both, are present in high concn.) Pontachrome violet SW as indicator. (From English summary.) W. J. BAKER

1967. **Determination of low levels of furazolidone in feeds.** L. R. Stone, P. A. Lewis, M. T. Miller and C. F. Berg (Hess and Clark Inc., Ashland, Ohio, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 615–618.—The official (A.O.A.C.) method (*Ibid.*, 1958, **41**, 51, 333) is modified. Complete extraction of the furazolidone is ensured by heating the pellets and dimethylformamide at 92° before shaking. By the use of an Al_2O_3 column adsorption step in the procedure much of the interfering colour can be removed. A. A. ELDRIDGE

1968. **Micro-determination of reserpine in feeds with nitrous acid.** R. P. Haycock, P. B. Sheth, R. J. Connolly and W. J. Mader (Res. Dept., CIBA Pharm. Products Inc., Summit, N.J., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 613–615.—Micro amounts of reserpine in feeding-stuffs are extracted by a selective procedure in which $CHCl_3$ in the presence of dilute acid and of $NaHCO_3$ soln. is used; the $NaHCO_3$ removes inhibiting substances, reserpic acid and trimethoxybenzoic acid. The reserpine is finally determined by the nitrite method (Banes, *Anal. Abstr.*, 1958, **5**, 2366). A. A. ELDRIDGE

1969. **Determination of rotenone in lonchocarpus root and oleo-resin.** D. V. Richmond and J. T. Martin (Dept. of Agric. and Horticulture, Long Ashton Res. Station, Univ. Bristol, England). *J. Sci. Food Agric.*, 1959, **10** (7), 404.—The determination of rotenone (**I**) by direct crystallisation of a CCl_4 solvate gives low results because of impurities which retard crystallisation. Yields of **I** after treatment of the extract with alkali or by percolation through activated alumina were up to 18% higher than those obtained by direct crystallisation. Recovery of pure **I** from an alumina column was 96·5%; the loss on the column itself was about 2·5%. P. D. PARR-RICHARD

1970. **Quantitative determination of arsenic residues in plant materials.** H. Frehse and H. Tietz (Biol. Inst., Farbenfabrik. Bayer A.-G., Leverkusen, Germany). *J. Agric. Food Chem.*, 1959, **7** (8), 553–558.—Arsenic in plant material is determined by wet-ashing, adding KBr, and distilling in a special apparatus (illustrated), fitted with a trap in which the $AsBr_3$ collects. Arsenic is measured as the molybdenum blue complex, preferably at 840 mμ in a 5-cm cell. From 1 to 60 μg of As per sample can be determined by direct reading from a standard curve. When applied to apples 4 to 14 weeks after the last of 10 applications of a spray containing As, the method showed residues of up to 0·25 p.p.m. of As. M. D. ANDERSON

1971. **Separation and identification of organic mercury compounds by paper chromatography.** Jun Kanazawa and Rokuro Sato (Agric. Chem. Inspection Station, Nishigahara, Kita-ku, Tokyo). *Japan Analyst*, 1959, **8** (5), 322–323.—For the paper-chromatographic separation of alkyl- and aryl-mercury compounds used as agricultural chemicals, the use of a mixture of *n*-butanol, pyridine and *N* aq. NH_3 soln. (35:34:31) is recommended. The R_F values are as follows—*p*-tolylmercury acetate, 0·77; phenylmercury acetate, 0·68; di-(ethylmercury) phosphate, 0·62; 2-methoxy-ethylmercury chloride, 0·47; methylmercury chloride, 0·41; $HgCl_2$, 0·06. The spots are revealed with 1% diphenylcarbazone soln. in ethanol. K. SAITO

1972. **The determination of fluoroacetic acid and fluoroacetamide in plant material.** C. C. Thompson (Min. of Agric., Fish. & Food, Plant Path. Lab., Harpenden, England). *J. Sci. Food Agric.*, 1959, **10** (7), 388–393.—Fluorides and fluorosilicates are removed by treatment with silica gel (*cf.* Ramsay and Clifford, *J. Ass. Off. Agric. Chem.*, 1949, **32**, 788) before the determination of fluoroacetic acid (**I**). After extraction of the percolate with diethyl ether, the soln. is evaporated in the presence of CaO, charred and ashed at 600° and steam-distilled in the presence of silver perchlorate and $HClO_4$. The neutralised distillate is again evaporated and ashed with CaO and the distillation is repeated. Fluoride in the distillate is determined with thorium - alizarin reagent (*cf.* Icken and Blank, *Anal. Abstr.*, 1954, **1**, 695). The accuracy for 1 p.p.m. of F^- is within ≃10% and for 0·1 p.p.m. is ≃50%. The recovery of added fluoroacetate was from 86 to 95%. To determine fluoroacetamide (**II**), acid radicals are removed with De-Acidite FF anionic resin. The percolate containing **II** is boiled with NaOH, the soln. is evaporated with CaO, and **I** is determined as before. Present evidence indicates that little hydrolysis of **II** to **I** occurs in plants. P. D. PARR-RICHARD

1973. **Micro-bioassay of insecticide residues in plant tissues without extraction, with special reference to aldrin and dieldrin.** N. W. Earle, J. E. Pankaskie and Yun-Pei Sun (Entomol. Dept., Shell Development Co., Modesto, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 586–592.—Adult pomace flies, *Drosophila melanogaster* Meig., are exposed to macerated plant tissues, and the mortality resulting from contact with and ingestion of the insecticide (which may have a relatively low solubility in water) is compared with that resulting from exposure to standards. As little as 0·1 p.p.m. of aldrin or dieldrin in various fruits and vegetables could thus be detected. Extraction methods have the advantage of permitting increased sensitivity. A. A. ELDRIDGE

1974. **Colorimetric method for determination of Dyrene [2:4-dichloro-6-o-chloroanilino-*sym.*-triazine] residues in plant material.** W. R. Meagher, C. A. Anderson, C. E. Gonter, S. B. Smith and D. MacDougall (Res. Dept., Chemagro Corp., Kansas City, Mo., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (8), 558–560.—Residues of this fungicide (**I**) are determined by blending the sample with *iso*propyl alcohol, extracting the **I** into benzene, evaporating the solvent, hydrolysing the residue with acid to give *o*-chloroaniline, reducing with zinc to diminish extraneous colour, adding Celite and filtering, diazotising, and coupling with N-1-naphthylethylenediamine to produce a magenta colour, which is measured at 540 mμ. The limit of sensitivity of the method is 1·0 p.p.m., which is satisfactory in view of the low toxicity of **I**. Mean recoveries of **I** added to celery, onions, potatoes and tomatoes were 84 to 103%. M. D. ANDERSON

1975. **Determination of Perthane [1:1-dichloro-2:2-di-(*p*-ethylphenyl)ethane] and mixed Perthane - DDT residues.** F. J. Roth (Bur. Chem., State Dept. Agric., Sacramento, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 607–609.—Similarly to compounds that produce colour in the abridged Schechter procedure, *e.g.*, DDT (*cf.* Pontoriero and Ginsburg, *Anal. Abstr.*, 1954, **1**, 1992), 1:1-dichloro-2:2-di-(*p*-ethylphenyl)ethane (**I**) can be nitrated and the product dissolves in benzene, but it differs from DDT, DDD [dichlorodi(chlorophenyl)ethane], methoxychlor, chlorobenzilate and Kelthane [2:2:2 - trichloro - 1:1 - di - (*p* - chlorophenyl)-ethanol] in that it does not then produce a colour with Na methoxide. However, colour is developed by heating the nitrated product under reflux with *N* ethanolic KOH and subsequently acidifying. The procedure for the preparation of standard curves for **I** and DDT and for the analysis of a mixture of the two insecticides is described.

A. A. ELDRIDGE

1976. **The behaviour of chlorine-bearing organic compounds in the A.O.A.C. available chlorine germicidal equivalent concentration test.** L. F. Ortenzio and L. S. Stuart (Pesticide Reg. Sect., Agric. Res. Service, U.S. Dept. Agric., Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (3), 630–633.—Tests with 1:3-dichloro-5:5-dimethylhydantoin, dichloro*iso*cyanuric acid and trichloro*iso*cyanuric acid show that results obtained with chloramine T in the A.O.A.C. available Cl germicidal equivalent concentration test ("Official Methods of Analysis," A.O.A.C., 8th Ed., 1955, p. 93) are not an accurate guide to their germicidal activities. Curves show their concentrations (in p.p.m. of available Cl) which at pH 6 to 10 are equivalent to hypochlorite soln. at pH 8·5 providing 200, 100 and 50 p.p.m. of available Cl. Satisfactory formulations are indicated.

A. A. ELDRIDGE

1977. **Determination of Alodan residues in grain.** G. Paulig (Max von Pettenkofer Inst., Abt. f. Lebensmittelchemie, Berlin-Dahlem). *Z. anal. Chem.*, 1959, **168** (6), 401–406 (in German).—The method is based on the formation of dimethylene-hexachlorobi*cyclo*heptene (**I**) by treatment of Alodan [1:2:3:4:7:7-hexachloro-5:6-bis(chloro-methyl)bi*cyclo*[2:2:1]hept-2-ene] with ethanolic KOH. The grain (500 g) is extracted with 700 ml of CCl_4 for 30 min. and the filtered extract is evaporated to 50 ml on a water bath at 85°. The concentrated extract is filtered, dried over Na_2SO_4 and chromatographed on an alumina column. The column is eluted with 200 ml of CCl_4 and the eluate is evaporated to dryness; coloured impurities remain on the first 4 cm of the column. The residue from the eluate is dissolved in 25 ml of abs. ethanol and heated to 85°, at which point 100 ml of ethanolic 2% KOH soln. is added and the temp. is maintained for a further 30 min. At least 500 ml of water is then added and the mixture is extracted in three stages, each with 50 ml of *cyclo*hexane that has previously been purified by fractional distillation. The combined extracts are washed (preferably with satd. aq. NaCl soln. to prevent emulsion formation), dried and evaporated, and the residue is dissolved in 5 ml of CS_2. The i.r. spectrum of the soln. is measured between 11 and 15 μ, and the concn. of **I** is determined from the band at 13·28 μ. Amounts down to 1 p.p.m. can be determined with an error of $<5\%$; the error is smaller (1%) for more than 2 p.p.m. By scaling-up the procedure and taking 1 kg of grain, concn. of 0·5 p.p.m. can be measured.

S. M. MARSH

1978. **The micro-estimation of γ-(4-chloro-2-methylphenoxy)butyric acid [MCPB], γ-(2:4-dichlorophenoxy)butyric acid [2,4DB] and *n*-butyl γ-(2:4-dichlorophenoxy)butyrate in plant material.** H. A. Glastonbury and M. D. Stevenson (Res. Lab., May and Baker Ltd., Dagenham, England). *J. Sci. Food Agric.*, 1959, **10** (7), 379–385.—The determination of phenoxyalkanecarboxylic acids and ester residues in plants gives low results which are corrected for by an isotopic dilution technique. Some results obtained in field work are given. *Procedure for MCPB and 2,4DB*—Dry 50 to 200 g of plant material at 100°, make into a slurry with 100 ml of 0·1 *N* NaOH, add 20 μg of ^{14}C-labelled MCPB or 2,4DB and set aside for 1 hr. Filter and wash the residue with H_2O (100 ml), acidify the filtrate with 10 ml of conc. HCl, boil and cool. Add an excess of conc. aq. tungstophosphoric acid soln., set aside for 15 min. and filter. Extract the filtrate with CCl_4 and filter the solvent layer. Extract this with 5% $NaHCO_3$ soln., acidify the aq. layer, boil and cool. Extract with *n*-hexane, filter the extract and evaporate to dryness; add 3 ml of dry acetone and set aside for 10 min. Add the soln. in small amounts to 300 mg of KBr, air-dry, and mix. Prepare a disc and measure the i.r. absorption at 13·5 μ for 2,4DB and at 15·15 μ for MCPB. Dissolve the disc in *N* HCl, extract with CCl_4 and evaporate to dryness. Dissolve the residue in methanol (5 ml). Place 0·1 ml of this soln. and 0·1 ml of 0·1% aq. agar soln. on a counting planchette, evaporate gently, and measure the activity. *Procedure for the butyl ester of 2,4DB*—Mix 25 to 50 g of plant material with water (200 ml). Add 20 μg of ^{14}C-labelled ester and steam-distil. Extract 600 ml of distillate with diethyl ether and evaporate to dryness. Add 25 ml of 0·1 *N* NaOH and 50 ml of methanol, boil under reflux for 30 min. and distil off the methanol. Dilute the soln. to 100 ml, acidify with HCl and extract with CCl_4. Proceed as for MCPB and 2,4DB.

P. D. PARR-RICHARD

See also Abstracts—**1677**, Determination of ^{89}Sr and ^{90}Sr in soil. **1681**, Zinc in plant ash. **1695**, Silica in soil and plants. **1829**, Starch in vegetable matter. **1843**, Calcium in plants. **1851**, Carotene in grass. **1875**, 5-Vinyl-2-oxazolidinethione in plants. **1913**, Fat in feeding-stuffs.

5.—GENERAL TECHNIQUE AND APPARATUS

General

1979. **Possible source of error arising in analysis from the use of polyethylene bottles.** L. S. Theobald (Chem. Dept., Imperial Coll. of Science and Technology, London). *Analyst*, 1959, **84**, 570–571.—Attention is drawn to the absorption and subsequent slow release of H_2S, NH_3, Br and HNO_3 by polyethylene wash-bottles. With H_2S and Br, many fillings with water, with intervening periods of setting aside the empty bottles, were necessary to remove the contamination. The removal is helped by passage of a current of air. This permeability of polyethylene apparatus to certain gases and vapours may be an unsuspected source of error in analytical work.

A. O. JONES

1980. **Automatic moisture indicator [for clay].** M. E. C. Stedham (Western Counties Brick Co. Ltd., Exeter, England). *Trans. Brit. Ceram. Soc.*, 1958, **57** (7), 381–392.—In the apparatus described and

illustrated, the changes in weight on heating successive samples (20 to 30 g) of ground clay in a rotating copper cylinder are converted by an electrical circuit into percentages of water (dry basis). These values are recorded on a chart at intervals of ≃6 min. After calibration of the instrument, the error is <0·5% for moisture contents of 4 to 12%. All operations, including the collection of samples from the moving stream of clay, are carried out mechanically. W. J. BAKER

1981. **Simple circuit for the determination of the end-point by the dead-stop method.** J. F. W. Tertoolen (Anal. Inst. T.N.O., Rijswijk, Netherlands). *Chem. Weekbl.*, 1959, **55** (23), 270.—A circuit diagram is given in which the use of a sensitive and expensive light-spot galvanometer is avoided. Transistors are used as amplifiers and a simple micro-ammeter is used as a zero-point instrument. The arrangement has been applied to the titration of manganese according to Lingane and Karplus and to the titration of iodine with thiosulphate. M. J. MAURICE

1982. **Method of and apparatus for gas analysis.** Hartmann & Braun A.-G. Brit. Pat. 824,854, date appl. 5.4.56. Germany, date appl. 6.4.55.—The proportion and calorific value of the unburnt constituents of flue gases, protective gases, etc., are determined by submitting the gases (possibly after admixture of a predetermined quantity of O or of an O-containing gas of known O content) in a reaction chamber to a reaction in which their reactable constituents bind or liberate O. The O concn. in the gas being tested (or its mixture with an O-containing gas) is determined, before and after the reaction, in a paramagnetic oxygen meter in which the flowing gases are exposed, in a thermal-conductivity cell, to a constant inhomogeneous magnetic field. The difference between the two concn. is used as a measure for the determination of the calorific value of the combustible constituents and/or of the proportion of reducing or otherwise reactable constituents of the gas. J. M. JACOBS

1983. **Improvements in or relating to gas-analysis apparatus.** Mine Safety Appliances Co. [Inventor: J. L. Waters]. Brit. Pat. 807,437; date appl. 31.7.57.—In the analysis of gases by existing methods, it is desirable that the sample should be at a predetermined temp. and pressure, and hence at a predetermined density. Details are given of a heat-exchange chamber, with a porous partition of sintered stainless steel, for supplying gas at a predetermined temp. to an i.r. gas-analysis apparatus. J. M. JACOBS

1984. **Improvements in the separation of mixtures in the presence of a carrier gas.** Badische Anilin- & Soda-Fabrik A.-G. Brit. Pat. 824,297; date appl. 23.3.56. Germany, date appl. 26.3.55.—The vaporisable components resulting from (exothermic) chemical reactions are separated by entraining the mixture in a carrier gas, which is not condensable at the temp. and pressure used in the process, and bringing the vapour-laden gas into contact, in an exchange device, with a liquid obtained by condensation of the vapours from the carrier gas. The less volatile components are thus removed in condensed form and the more volatile components are removed in vapour form, together with the gas. J. M. JACOBS

1985. **Washing apparatus of high capacity for very slightly soluble chemical products.** M. R. H. Rigot (Cie. Gén. de T.S.F., France). *Chim. Anal.*, 1959, **41** (7), 296–298.—The apparatus described is based on the Soxhlet principle: water is distilled from a 5 to 6-litre flask carrying a gas inlet, through a wide tube by-passing the washing vessel, to a condenser so constructed as to allow a stirrer to pass through it into the washing vessel. The water from the condenser drips into this cylindrical 2-litre vessel, which has a sintered glass disc to retain the material being washed. The warm wash water from underneath the disc is siphoned into a water-jacketed vessel, at the bottom of which sealed-in electrodes enable the conductivity to be measured. From this cell the water is returned to the boiler. A small condenser is described which enables the conductivity of a small known quantity of distillate from the boiler to be determined at constant temp. in order that the best washing conditions may be determined. E. J. H. BIRCH

1986. **Apparatus for the determination of ammonia by micro-diffusion.** A. Mari, M. Talarico and E. Scarano (Ist. di Fisiol. Umana, Univ. di Napoli). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (13), 789–792.—The micro-apparatus consists of male and female parts which are fitted together as a tapered ground glass joint. The female part has two bulbs blown linearly on it: a larger one (30 mm in diam.) to hold the extract (containing 0·2 to 1·4 μmoles of NH_3), and a smaller distal bulb (18 mm in diam.) holding 0·2 ml of 3 *M* NaOH. On the male joint is blown a single bulb (30 mm in diam.) holding 0·1 ml of 1 *N* HCl. After the two halves are fitted together, 2·5 to 3 hr. is sufficient to complete the distillation into the acid, the NH_3 in which is determined by nesslerisation. C. RAINBOW

1987. **Simple device for avoiding stoppages in distillations under reduced pressure.** H.-J. Haase (Forschungsabt. d. VEB Berlin-Chem.). *Chem. Tech., Berlin*, 1959, **11** (7), 382.—To prevent condensations of solid distillate at constrictions in the vacuum line, the vapour passes through a wide tube (fitted with a bulb) from a three-necked distillation vessel containing a non-volatile solvent, the end of the tube being <2 cm above the liquid surface. The other two necks are connected to the vacuum line and are fitted with taps to the atmosphere. The device has been useful in distilling α-chloromethylnaphthalene containing naphthalene. A. R. PEARSON

1988. **Direct-reading viscometer.** M. R. Shafer. *Instrum. and Control Systems*, 1959, **32** (7), 1044–1047.—An instrument based on the restrictions-in-series principle, and suitable for the determination of viscosities over the range 0·5 to 5·0 centistokes with an accuracy of 2%, is described. G. SKIRROW

Chromatography, ion exchange, electrophoresis

1989. **Increasing the rate of elution in chromatographic columns.** L. Westenberg (Plantenziektenkundige Dienst, Wageningen, Netherlands). *Chem. Weekbl.*, 1959, **55** (31), 410.—To increase the rate of elution it is often recommended to exert pressure on the eluent by means of an inert gas. However, after a time channelling occurs in the filling of the column which prevents a regular flow of the eluent. This disadvantage can be overcome by using the hydrostatic pressure of the eluent instead of gas pressure. M. J. MAURICE

1990. **Simple [chromatographic] fraction collector.** H. Wolffgang and R. Seiler (Biol. Zentralanst., Dtsch. Akad. d. Landwirtschaftswiss., Berlin). *Chem. Tech., Berlin*, 1959, **11** (7), 381–382.—The eluate is received in a test-tube carried in a slightly sloping position on a shaft so that when filled to a given level it tilts and empties its contents through a funnel into one of 96 receivers fixed in a rotatable aluminium plate. The tube is held in its tilted position until empty by a sealed tube mounted on the same shaft and loaded with a steel ball. It is then returned by means of an electromagnet controlled synchronously with the rotation of the plate by a mercury time-switch. A. R. PEARSON

1991. **Horizontal chromatography.** F. Savoia (Lab. Ter. Sper. "Bruschettini," Genova, Italy). *Farmaco, Ed. Prat.*, 1959, **14** (7), 424–427.—A detailed description is given of the equipment and technique used for carrying out serial chromatographic separations of mixtures of substances on paper held horizontally. L. ZANONI

1992. **Paper chromatography by means of a "line oven."** A. Biró (Inst. for Inorg. and Anal. Chem., Sci. Univ., Szeged, Hungary). *Magyar Kém. Foly.*, 1959, **65** (7), 245–249.—The method is based on the ring-oven technique, but the material from the drop applied to the paper is concentrated in a smaller area, as a line, so that the sensitivity of the test reactions is increased and components present in amounts of 10^{-6} to 10^{-7} g can be separated on one strip. By using a series of reagents and a double oven, a mixture of ions can be separated into its constituents. As an example, the separation (in 30 to 40 min.) of the following ions from 0·005 ml of a soln. is described—Ag, Hg, Cu, Sn, Ni, Fe, Cr, Ca, Sr and Ba. The process and the apparatus are described in detail. A. G. PETO

1993. **Constant-temperature explosion-proof cabinet for development of two-dimensional chromatograms.** J. Logothetis and H. P. Cohen (Neurol. Div., Univ. Med. School, Minneapolis, Minn., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1601–1602.—The apparatus consists of a double-walled steel chromatographic cabinet, which is electrically heated and thermostatically controlled. At the same time, warm air is made to circulate in the space between the two walls, which is partly filled with glass wool. Excellent temp. control is afforded. For a working temp. of $\simeq 31°$, the temp. variation in the cabinet is $\gtrless 0{\cdot}32°$.
J. H. WATON

1994. **Quantitative evaluation of paper chromatograms by reflectance measurements.** G. Kortüm and J. Vogel (Inst. phys. Chem., Univ., Tübingen). *Angew. Chem.*, 1959, **71** (14), 451–455.—The Kubelka - Munk relationship is not strictly applicable to the quant. evaluation of paper chromatograms by reflectance measurements because the dye is not uniformly distributed on the fibre. By carrying out measurements on paper impregnated with malachite green (C.I. Basic Green 4) it has been found that quant. results can be obtained by working with very small quantities of dyestuff, and with diffuse illumination, ensuring that the paper always contains a fixed amount of water during reflectance measurement and by using a special background consisting of ten layers of white paper. W. T. CARTER

1995. **Gas-phase chromatography. Proposals for a vocabulary and system of notation for retention values.** J. Buzon, P. Chovin, L. Fanica, P. Ferrand, G. Guiochon, M. Huguet, J. Lebbe, J. Serpinet and J. Tranchant. *Bull. Soc. Chim. France*, 1959, (7–8), 1137–1140.—It is suggested that a nomenclature should be standardised for retention values (distance, time or volume), and it is proposed that the term "retention value" should be used for the measurement from the injection of the sample to the recording of the peak max.; the "reduced retention value" should be measured from the passage of the "air peak" through the recorder, the "limiting retention value" should be the retention value corrected by James and Martin's factor (*Biochem. J.*, 1952, **50**, 679), and the "absolute" or "limiting reduced" retention value should be the reduced retention value similarly corrected. "Relative retention values" are defined as the ratio of reduced or limiting reduced retention values for a substance and an internal standard. It is suggested that the exponent (°) should be used for limiting and (′) for reduced values, *e.g.*, "limiting reduced retention volume" is expressed by $(V_R°)'$. It is suggested that symbols should be chosen with a view to use in all languages, and a list of suggested symbols is given.
E. J. H. BIRCH

1996. **Improvements relating to vapour-phase chromatography.** Metropolitan-Vickers Electrical Co., Ltd. [Inventor: G. Burrows]. Brit. Pat. 818,703; date appl. 27.3.56.—Apparatus for vapour-phase or gas chromatography comprises a series of parallel ducts, for containing the porous adsorption material, extending between an inlet header, to which inert gas will be supplied, and an outlet header, with an outlet passage through which vapour separated in the ducts can leave with the inert gas. Injectors are provided in the inlet header for injecting into the ducts a charge of a fluid mixture the components of which it is desired to separate.
J. M. JACOBS

1997. **Automatic analysis of flowing gases by means of the chromatograph KhPA-1.** A. I. Tarasov, N. A. Kudryavtseva, A. V. Ioganson and N. I. Lulova (All-Union Inst. of Petroleum and Gas Refining and Manufacture of Artificial Liquid Fuel). *Zavod. Lab.*, 1959, **25** (7), 803–805.—The apparatus and some of its applications are described. G. S. SMITH

1998. **Ionisation detector with promethium-147 for gas chromatography.** S. N. Oziraner, G. A. Gaziev, M. I. Yanovskiĭ and V. S. Kornyakov (Inst. of Phys. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1959, **25** (6), 760–761.—The use of ^{147}Pm as a source of β-radiation is described.
G. S. SMITH

1999. **Simplified fluid-film method of electrophoresis.** N. Ressler and T. Moy (Wayne County Gen. Hosp., Eloise, Mich., U.S.A.). *Clin. Chim. Acta*, 1959, **4** (6), 901–904.—The construction, use and results obtained with an apparatus that eliminates the use of filter-paper wicks for connection of the fluid film with the electrode vessels are described. H. F. W. KIRKPATRICK

Optical

2000. **Improvements in and relating to grating spectrometers or analysers.** Sir Howard Grubb Parsons & Co., Ltd. [Inventor: A. E. Martin]. Brit. Pat. 825,508; date appl. 21.9.56.—Spectrometers in which a diffraction grating is used to disperse incident radiation are provided with a

second grating for the elimination of at least the second order of diffraction from the first grating.
J. M. JACOBS

2001. **Recording spectrophotometers. I.** P. W. J. M. Boumans (Lab. v. Anal. Chem., Univ. Amsterdam, Netherlands). *Chem. Weekbl.*, 1959, **55** (27), 329–331.—This paper deals only with spectrophotometers operating in the u.v., the visible and the near i.r. regions of the spectrum. A survey is given of the principles underlying modern spectrophotometers, and the registration of absorption spectra is discussed.

II. P. W. J. M. Boumans. *Ibid.*, 1959, **55** (28), 350–353.—The construction of spectrophotometers operating according to double-beam two-cell methods is discussed.

III. P. W. J. M. Boumans. *Ibid.*, 1959, **55** (29), 375–377.—The construction of spectrophotometers of the double-beam one-cell type is described.
M. J. MAURICE

2002. **Use of gas-discharge apparatus as light source for the spectrographic analysis of inert gases.** G. I. Vidro, B. D. Luft and Yu. V. Matorin. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 212–214; *Ref. Zhur., Khim.*, 1958, (18), Abstr. No. 64,149.—For determining impurities in inert gases used for filling gas-filled vacuum tubes and thyratrons, the spectra emitted by these sources are photographed. A hole of diam. 1 mm is made in the anode of the tube; this does not affect its working. The light from the hole is projected on to the slit of glass spectrograph ISP-51, with camera F-270. Neon was detected in spectrally pure He in TG-IP tubes. Standards were prepared by filling tubes with a mixture of Ne and He, and could be used to determine the concn. of Ne quantitatively. Good agreement was obtained between the results of this direct method and the normal more cumbersome method (in a h.f. discharge). The procedure has been introduced in industry for checking the purity of He used for filling tubes.
C. D. KOPKIN

2003. **Improvements in or relating to frequency spectrum analysers.** Marconi's Wireless Telegraph Co., Ltd. [Inventor: S. W. H. W. Falloon]. Brit. Pat. 823,425, date appl. 1.11.56.—The analyser comprises means for translating the spectrum of frequencies to be analysed into pressure-waves in a system comprising a light-transmitting pressure-wave propagating medium, a dispersive pressure-wave refracting device in the medium extending across the paths of the waves, a mask with a narrow slit, means for directing light through the slit along the focal surface of the refracting device along paths which cross the paths of the pressure-waves, and a light-obstructing member spaced from the mask and so positioned and dimensioned as to prevent light which has passed through the slit from being seen from a predetermined view-point when the medium is undisturbed; when the medium is disturbed by the passage of pressure-waves through it, light which has passed through the slit will be seen from the view-point at positions dependent on the frequency of the waves.
J. M. JACOBS

2004. **Wavelength calibration of spectrophotometers.** I. K. Walker and H. J. Todd (Dominion Lab., D.S.I.R., Wellington, N. Zealand). *Anal. Chem.*, 1959, **31** (9), 1603–1604.—A table is given of spectral lines suitable for wavelength calibration below 600 mμ, using a Beckman mercury lamp. Approximate slit values for the recommended lines are given. For calibration above 600 mμ, suitable hydrogen, argon and mercury lines are tabulated.
K. A. PROCTOR

2005. **Simple auxiliary recorder for the Perkin-Elmer infra-red spectrophotometer, model 21.** L. A. van Dijck and L. M. Simons (Res. Lab. N.V. Organon, Oss, Netherlands). *Chem. Weekbl.*, 1959, **55** (31), 407–408.—The construction of an auxiliary recorder, which produces a smaller copy of the spectrum simultaneously with the main recorder, is described. The coupling between the two recorders is mechanical and the auxiliary recorder is fully independent of the main recorder. The spectra are reduced 10:3.
M. J. MAURICE

2006. **Use of the Beckman DU spectrophotometer in the 1·0 to 1·9-micron range.** J. P. Phillips (Chem. Dept., Univ. of Louisville, Ky., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1604.—Modifications involving the use of a germanium photodiode are described. The principal advantages of such a diode are its extreme instrument simplicity, low cost, and peak response in the 1·2 to 1·6-μ range.
K. A. PROCTOR

2007. **Near-infra-red spectroscopy with the Beckman DU spectrophotometer.** W. E. Shuler (Savannah River Lab., E. I. du Pont de Nemours & Co., Aiken, S.C., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1604–1605.—Modifications involving the use of lead sulphide cells are described.
K. A. PROCTOR

2008. **Sodium azide as an internal standard for quantitative infra-red analysis.** R. T. M. Fraser (Dominion Lab., D.S.I.R., Wellington, N. Zealand). *Anal. Chem.*, 1959, **31** (9), 1602–1603.—Sodium azide has a very strong peak at 2140 cm^{-1} and a weak peak at 1309 cm^{-1}, and only a short grinding time is required to produce constant standard to sample peak-height ratios for identical mixtures. Sodium azide can be used as an internal standard in both liquid paraffin mulls and potassium bromide discs.
K. A. PROCTOR

2009. **Cell arrangement for increased utilisation of infra-red spectrophotometers in process studies.** C. E. Day (Polychemicals Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1605–1606.—An arrangement is described whereby a 1-cm sample cell and a 10-meter, folded-path, gas cell can be incorporated into an i.r. spectrophotometer so that either cell can be used as required without any necessity for exchanging them manually. In this way components in a gas stream at levels of 50% and $<0{\cdot}01\%$ can be monitored without loss of instrument time.
K. A. PROCTOR

2010. **Recording photometers.** P. W. J. M. Boumans (Lab. v. Anal. Chem., Univ. Amsterdam, Netherlands). *Chem. Weekbl.*, 1959, **55** (30), 388–390.—The principles of absorbance recorders are discussed.
M. J. MAURICE

2011. **Comparative measurements with different colorimeters and photometers. Third addendum.** H. Pohl (Federal Inst. for Testing Materials, Berlin-Dahlem, Germany). *ChemikerZtg*, 1959, **83** (15), 513–515.—Two new commercial instruments (illustrated) are tested for reproducibility and utility on coloured soln. and glass-filters (*cf. Ibid.*, 1957, **81**, 785). The Elektrophot I covers the range from 360 to 700 mμ (band-width 30 to 80 mμ) and has two alkali-vacuum photocells (sensitive to red and blue, respectively). The reproducibility is better than $\pm 0{\cdot}7\%$ in the extinction range from 0·125 to 1·0.

Deviations from absolute extinction values in the red region are due to the excessive band-width of the interference filters. The spectral colorimeter covers the range from 400 to 700 mμ (band-width 20 mμ) and has a selenium photo-element. The reproducibility is better than ±0·6% in the extinction range from 0·125 to 1·0. In continuous use a rise in temp. changes the sensitivity and causes zero drift. Extinction values are not absolute, but the instrument is very simple to use and well suited for routine control purposes. J. P. STERN

2012. **Automatic apparatus for the Lundegårdh method of analysis.** J. E. Allan and O. E. Clinton (Rukuhia Soil Res. Sta., Hamilton, N. Zealand). *N.Z.J. Sci.*, 1959, **2** (2), 219–229.—An apparatus is described for the mechanisation of the spraying procedure in the Lundegårdh spectrochemical method of analysis. It consists essentially of a shutter, a sample-changing machine, and a plate-racking mechanism; 58 spectra can be recorded on a 4-in. plate in about 1·5 hr. and provision is made for the replicate spraying of samples and for the return of the plate-holder and turn-table to the starting position when the series has been completed. E. G. BRICKELL

2013. **Phosphorimetry.** Z. Holzbecher (Dept. Anal. Chem., School of Chem. Technol., Prague). *Chem. Listy*, 1959, **53** (7), 713–717.—The principles are described of a method, analogous to fluorimetry, based on the measurement of the intensity and decay of the phosphorescent radiation of solid transparent soln. of compounds in inorganic solvents, cooled with liquid nitrogen. The possibilities of the use of this method in analysis are discussed. J. ZÝKA

2014. **Improvements in or relating to the analytical testing of liquids.** H. Fuhrmann. Brit. Pat. 817,474; date appl. 1.2.56. Germany, date appl. 9.11.55.—An automatic apparatus is described for the analysis of liquids by the addition of indicators, buffers and complexing agents, *e.g.*, nitrilotriacetic acid and disodium alkylenediaminetetra-acetate. In a first measuring stage the indicator is added, and in a second measuring stage the other additives are introduced in known amounts to the tinted liquid after it has been transferred to a second measuring chamber. The variations in the shades of colour so produced are measured comparatively by a differential method with photocells. The technique is employed more particularly for determining the hardness of water or its SiO_2, phosphate, sugar, chlorine or ozone content. J. M. JACOBS

Electrical

2015. **Improvements in and relating to fluid analysis.** Union Carbide Corp. Brit. Pat. 826,174; date appl. 12.2.57. U.S.A., date appl. 13.2.56.—Apparatus for the continuous detection and quant. determination of one constituent in the p.p.m. range of a mixture of gases, comprises a hollow cylindrical electrode that can be rotated about a horizontal axis, and is partly immersed in a solution of a reagent selected for its ability to change in ionic content upon contact with a selected constituent of the gas mixture. The electrode is rotated at constant speed to form a continuous thin film of reagent upon its unimmersed surface. A stream of the gas mixture is brought into contact with this film, and when rotation of the electrode returns the film of treated solution to the body of reagent a dynamic concentration cell is completed between the rotating electrode and a stationary probe electrode immersed in the body of reagent solution. The electric potential developed between the electrodes serves as a quant. measurement of the concn. of the selected constituent of the gas mixture. J. M. JACOBS

2016. **Controlled-potential and derivative polarograph.** M. T. Kelley, H. C. Jones and D. J. Fisher (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.) *Anal. Chem.*, 1959, **31** (9), 1475–1485.—The various features of this instrument are discussed. Controlled-potential polarography is especially suitable for high resistance media and has advantages over conventional polarography. With the polarograph described it is possible to record instantaneous currents, successive peak currents, successive average currents, or the time derivative of the polarographic wave as a function of the potential of the polarised electrode with respect to the soln. K. A. PROCTOR

2017. **A square-wave polarograph.** Yoshikazu Yasumori (Yanagimoto Co., Honmachi, Higashiyama-ku, Kyoto). *Japan Analyst*, 1959, **8** (6), 361–366.—A square-wave polarograph was set up in accordance with Barker's scheme (*Analyst*, 1952, **77**, 685). By the use of the Cd^{2+} wave, it was found that the condenser current decays within 1/2000 sec., whilst the faradaic current decays within 1/400 sec. With a change in the time constant of the condenser coupling in the oscillation amplifier, the shape of the plateau of the square wave changes, and there is an optimum condition under which the condenser current is best eliminated. The sensitivity was increased to 2×10^{-7} *M* Cd in 0·5 *N* HCl. K. SAITO

2018. **The role of tervalent iron in pulse polarography.** B. Ya. Kaplan (Chem. Lab. of the Geological Dept. of the Central Regions). *Zavod. Lab.*, 1959, **25** (6), 669–673.—A peak pulse polarograph in which the vertical shifts due to the presence of Fe do not occur is described. The determination of Pb in ores in the presence of acid-soluble compounds of Sn and large amounts of Fe is satisfactory. G. S. SMITH

2019. **Attempts to increase the sensitivity of the polarographic method.** R. Kalvoda (Polarographic Inst., Acad. Sci., Prague). *Chem. Listy*, 1959, **53** (7), 689–704.—A survey is given of three main possibilities, namely (*i*) modification of indicator electrode, (*ii*) apparatus with an "alternating component" and (*iii*) methods based on the reduction and removal of metal cations from the soln. into the mercury electrode and the polarographic or oscillopolarographic registration of the dissolution of the amalgam so formed. (65 references.) J. ZÝKA

2020. **The Potentiograph E336, a recording automatic titration apparatus.** S. Wolf (Metrohm A.-G., Herisau, Switzerland). *ChemikerZtg*, 1959, **83** (15), 518–520.—The instrument combines a high-resistance recording potentiometer with a mechanical device coupling the syringe burette to the driving mechanism of the recorder; the amount of titrant expelled is thus proportional to the forward movement of the paper (370 mm per run). The titration proceeds uniformly at a pre-set rate between 0·08 and 5·0 ml per min. The accuracy is within ±0·2%. The instrument can be used for a variety of acid - base, halogen or sulphide determinations and also as a pH and mV meter. J. P. STERN

2021. **Voltammetric, potentiometric and amperometric studies with a rotated aluminium-wire electrode. II. The R.Al.E. as indicator electrode in potentiometric and amperometric acid - base titrations.** I. M. Kolthoff and C. J. Sambucetti (Sch. of Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (2), 155–165.—The effect of F^- on the potential of the R.Al.E. is reported for a number of buffer soln. In general, within the range pH 1 to pH 6, the potential becomes 30 mV more negative per unit increase in pH at constant F^- concn., but, at a fixed pH in this range, changing the F^- concn. from 10^{-4} to 10^{-3} *M* results in a potential more negative by 100 mV. In phosphate buffers from pH 6 to pH 8 no variation of potential with F^- concn. was recorded, nor was such variation observed in a range of assorted buffer soln. from pH 8 to pH 12. Reproducible potentials, dependent on the F^- concn., were recorded in a barbitone buffer at pH 7. Potentiometric and amperometric acid - base titrations can be made with the R.Al.E., and two examples are quoted. T. R. ANDREW

2022. **Stable apparatus for high-frequency analysis.** Kō Ishii, Shōichi Hayashi and Shizuo Fujiwara (Univ. of Electro-Communications, Chofu, Tokyo, Japan). *Anal. Chem.*, 1959, **31** (9), 1586–1589.—General considerations of high-frequency analysis and its applications are discussed. Changes in the sample are detected as measurable responses of current and frequency shift. The photochemical reactions in the leaves of plants have been investigated. K. A. PROCTOR

2023. **Description and use of a high-frequency titration apparatus.** R. Morpain and S. Courtecuisse (Inst. Nat. de Rech. Chim. Appl., France). *Chim. Anal.*, 1959, **41** (7), 290–295.—A high-frequency titrimeter is described in which a quartz crystal (≃3 Mc/s) is connected to the grid of a triode valve with an oscillating anode circuit. The cell consists of a glass cell fitting into two copper rings which form a condenser in parallel with that of the anode circuit. The variations of current in the circuit are measured after amplification by a valve voltmeter. Examples of titration curves are given with reagents at concn. of 0·1 *N* to 0·001 *N*, for acid - base and pptn. aq. titrations. For acid - base titrations in glacial acetic acid the method is stated to be satisfactory. E. J. H. BIRCH

2024. **Continuous coulometric analysis using a working electrode of predetermined potential.** E. L. Eckfeldt (Leeds & Northrup Co., Philadelphia 44, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1453–1460.—The method described is suitable for the continuous measurement of low concn. of electro-oxidisable or reducible substances in flowing streams. The sample soln. flows through a cell in contact with a working electrode of large area, the potential being chosen so as to cause an electrochemical reaction with one species, but not, as far as possible, with other species in the soln. K. A. PROCTOR

2025. **Equipment for handling millicurie amounts of radio-isotopes.** W. J. Blaedel and E. D. Olsen (Chem. Dept., Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1608.—The equipment described is for use in removing samples from isotope bottles. It provides a minimum of 1 in. of lead shielding on all sides of the bottle while allowing positive control of the bottle during sample withdrawal. A simple device for removal and replacement of the bottle screw-cap is described. K. A. PROCTOR

2026. **Measurement of radio-isotopes by a windowless counter and comparison of the results with the window Geiger - Müller tube.** J. Klumpar, M. Majerová and P. Jiroušek (Inst. Nucl. Res., Acad. Sci., Prague). *Jaderná Energie*, 1959, **5** (6), 190–193.—The description and properties of a 4π flow counter are given. Corrections for the thickness of the supporting membrane for radio-isotopes of various β-radiation energies were determined, and the results of measurements of the activity were compared with those obtained with a Geiger - Müller tube. J. ZÝKA

2027. **Improvements in or relating to the electrophoretic separation of isotopic ions.** C. J. Van Oss and N. R. Beyrard-Benchemoul. Brit. Pat. 826,278; date appl. 8.4.57. France, date appl. 12.4.56.—A long, narrow band, formed by a membrane of close-grained porous material (*e.g.*, regenerated cellulose or collodion) that is capable of swelling in an electrolyte into a solid coherent gel having intercommunicating pores, the inner dimensions of which are slightly larger than the largest ions to be separated, is subjected to a longitudinal d.c. The band is impregnated with a conducting buffer soln. and small quantities of a soln. containing the isotopic ions to be separated are supplied periodically to one end of the band and portions of the buffer soln. are extracted from the other end. J. M. JACOBS

2028. **Analysis of thin metal films by neutron activation.** B. A. Thompson (General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1492–1495.—Methods are described for the quant. determination of 11 elements on a single sample weighing ≃50 μg. Two chemical separation schemes, one for Cu, Cd, Sn, Mo, W, Cr, Ni and Fe, and one for Zn, P and Ta, are given. These schemes may be modified for the determination of other elements. The over-all precision is about ±4%. K. A. PROCTOR

2029. **High-mass spectrometry. Propylene polymer, alkylated benzene and wax analysis.** R. A. Brown, D. J. Skahan, V. A. Cirillo and F. W. Melpolder (Atlantic Refining Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1531–1538.—Improvements in high-mass spectrometric analysis, giving greater reliability and versatility, are described. The reproducibility of spectra and the establishment of methods of calculation were studied and applied to the analysis of propylene polymer, alkylated benzene and waxes. The accuracy for the polymer and alkylated benzene determinations were ±2 to 3% and ±1%, respectively. G. P COOK.

2030. **A 180° mass spectrometer for analysis of organic compounds of high molecular weight.** R. Ryhage (Lab. for Mass Spectrometry, Kem. Inst. I, Karolinska Inst., Stockholm). *Ark. Kemi*, 1959, **13** (5), 475–484 (in English).—The apparatus (described and illustrated) has a heated inlet system to effect complete vaporisation of the sample. Mass spectra have been determined of compounds with mol. wt. up to 619. A complete spectrum may be obtained with a sample of ≃0·5 μmole. The unit resolution extends up to *m/e* 500. A. G. COOPER

See also Abstract—**1981,** Dead-stop end-point determination.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	$R_{\mathbf{F}}$
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$
not greater than	$\ngtr$
is proportional to	$\propto$
less than	$<$
not less than	$\nless$
of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

Volume 7. No. 5. Abstracts 1637–2030 May, 1960

ANALYTICAL ABSTRACTS

A PUBLICATION OF THE SOCIETY FOR ANALYTICAL CHEMISTRY

EDITORIAL COMMITTEE

Chairman: B. A. Ellis

Members: B. S. Cooper, C. H. R. Gentry, C. A. Johnson, H. F. W. Kirkpatrick, B. J. Walby, W. A. Waygood, K. A. Williams and

President of the Society: R. C. Chirnside

Hon. Secretary of the Society: R. E. Stuckey

Hon. Treasurer of the Society: A. J. Amos

Editor: Norman Evers, B.Sc., Ph.D., F.R.I.C.

Assistant Editor: Mrs. H. I. Fisk, B.Sc.

CONTENTS

	Abstract
General Analytical Chemistry	
Reviews; reagents; methods of general application	1637
Inorganic Analysis	
General methods; elements (in order of the Periodic Table); minerals; industrial products	1654
Organic Analysis	
Elements and radicals; organic compounds; industrial products	1770
Biochemistry	
BIOLOGICAL FLUIDS; ANIMAL AND VEGETABLE TISSUES	1841
PHARMACEUTICAL ANALYSIS	1884
FOOD	
Food additives; beverages; edible oils and fats; vitamins	1910
SANITATION	
Air; water; sewage; industrial wastes	1949
AGRICULTURAL ANALYSIS	
Soil; fertilisers; herbicides; pesticides; animal feeding-stuffs	1960
General Technique and Apparatus	
GENERAL	1979
CHROMATOGRAPHY; ION EXCHANGE; ELECTROPHORESIS	1989
OPTICAL	2000
ELECTRICAL	2015

Printed and published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England.
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1.
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office.